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Trends of Perfluoroalkyl Acid Content in Articles of Commerce

— *Market Monitoring from 2007 through 2011*

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Table of Contents

List of Tables

List of Figures

Abstract

The U.S. Environmental Protection Agency (EPA) has established an ongoing effort to quantify possible changes in levels of perfluorinated chemicals (PFCs) in articles of commerce (AOCs). Temporal trends in the concentrations of selected PFCs, including perfluorooctanoic acid (PFOA) and other perfluorocarboxylic acids (PFCAs), in 35 AOCs were measured from the year of 2007 through 2011. The AOC samples that were collected included carpet, commercial carpet-care liquids, household carpet/fabric-care liquids, treated apparel, treated home textiles, treated non-woven medical garments, floor waxes, food-contact paper, membranes for apparel, and thread-sealant tapes. They were purchased from retail outlets in the United States between March 2007 and September 2011. Two to five AOCs from each of the ten categories were monitored. Depending on the market availability, products were collected for two to four data points in a span of four years. The perfluorocarboxylic acid (PFCA) contents in AOCs have shown an overall downward trend as portion of the fluorochemical industry has reformulated their PFC products. However, PFOA (C8) could still be detected in many AOCs that we had monitored. No obvious tendencies for change over the monitored period for short-chain PFCA (sum of C4 to C7) versus long-chain PFCA (sum of C8 to C12) were observed. A longer and wider range of monitoring will be required to confirm an observed trend. In addition to monitoring PFCA contents in AOCs, fourteen AOC samples were analyzed to determine the amounts of perfluoroalkyl sulfonates (PFAS) they contained. The limited data show the pronounced increase of perfluoro-butane sulfonate (PFBS-C4), an alternative to perfluorooctanoic sulfonate (PFOS), in the samples.

Acronyms and Abbreviations

AOC	articles of commerce
BDL	below detection limit
CAS#	chemical abstract service registration number
CFR	Code of Federal Regulations
DCC	daily calibration check
DQI	data quality indicator
EPA	Environmental Protection Agency
HPLC	high-performance liquid chromatography
IAP	internal audit program
IDL	instrument detection limit
IS	internal standard
LC/MS/MS	liquid chromatography/tandem mass spectrometry
MDL	method detection limit
NRMRL	National Risk Management Research Laboratory
NIST	National Institute of Standards and Technology
OPPT	Office of Pollution Prevention and Toxics
PFAS	perfluoroalkyl sulfonates
PFC	perfluorochemical
PFCA	perfluorocarboxylic acid
PFOA	perfluorooctanoic acid
PFOS	perfluorooctane sulfonate
QC	quality control
RCS	recovery check standard
RSD	relative standard deviation
SNURs	Significant New Use Rules
TSCA	Toxic Substances Control Act
TPFCA	total perfluorocarboxylic acids (the concentration sum of C4 to C12, C6 to C12 or C8 to C12 PFCAs)
TPFAS	total perfluoroalkyl sulfonates (the concentration sum of C4, C6, C7, C8, and C10 PFAS)

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1. Introduction

Perfluoroalkyl acids (PFAAs) came to the attention of researchers and risk managers because of their persistence, developmental toxicity and other health effects in laboratory animals [1, 2] and their ubiquitous presence in humans, wildlife, and environmental media. [3-10] To fully understand the health and environmental risks associated with PFCAs and related chemicals, EPA's Office of Pollution Prevention and Toxics (OPPT) was interested in investigating the role that articles of commerce (AOC) containing or having been treated with fluoropolymers and fluorotelomers have in human exposure in the microenvironments of homes and offices and as a source of environmental exposure once released to the outside world.

A project to test the perfluorocarboxylic acid (PFCA) content of various AOCs began in late 2006 in response to OPPT's need for data for use in assessing the risk of human exposures to perfluorooctanoic acid (PFOA) and other PFCAs. In March 2007, in Phase 1 of the project, EPA's National Risk Management Research Laboratory (NRMRL) initiated the collection of AOC samples in determining the PFCA content in new AOCs, and the analyses of all the samples were completed in May 2008. [3, 11] In that phase of the project, 13 categories of articles containing 131 consumer articles produced in 19 countries were collected. These articles were believed to have been treated with fluorinated chemicals and were analyzed to determine their content of five to twelve carbons (C5 to C12) PFCAs. We analyzed 116 of the 131 samples successfully and found that their total PFCA concentrations (i.e., the sum of C5 through C12) ranged from non-detectable to 47,100 ng/g, whereas their PFOA concentrations ranged from non-detectable to 6,750 ng/g. One of the major findings in Phase 1 was that, among the 13 article categories, commercial carpet-care liquids, mill-treated carpeting, treated floor waxes and sealants, and treated home textile and upholstery were potentially the largest PFCA sources in non-occupational indoor environments. The results of Phase 1 provided a snapshot of the transition period during which the use of fluorotelomer and fluoropolymer products in consumer products was changing rapidly. The limited data from the Phase 1 study suggested that some fluorinated surface-modifying agents had been reformulated to lower the PFCA content. The trends were uneven and many articles with high PFOA content could still be found on the market.

In May 2000, 3M, the primary American producer of perfluorooctane sulfonate (PFOS), announced the phase-out of its production of PFOS. However, there may still be other producers of PFOS-related compounds around the world.

In 2006, eight major companies in the PFC industry and the EPA jointly launched the PFOA Stewardship Program. Under this cooperative program, the goal is to reduce facility emissions to all media of PFOA, precursor chemicals that can break down to PFOA, and related higher homologue chemicals and product content levels of these chemicals by 95 percent by 2010, and to work toward the elimination of these chemicals from emissions and products by 2015. [12] Also, EPA promulgated three Significant New Use Rules (SNURs) under the Toxic Substances Control Act (TSCA) to limit any future manufacture or importation of 271 perfluoroalkyl sulfonates (PFAS) [13]. Thus, as portion of the fluorochemical industry reformulates its products, it is

anticipated that the overall content of PFOA and other PFCAs in articles of commerce will show a downward trend. However, it will take a long time (i.e., several years) and extensive sampling to verify if this indeed is a significant trend.

The project to test for PFCAs in AOCs was extended into a second phase to assess the market trends between 2007 and 2011. The objectives were to determine how the levels of PFCAs in AOCs changed over the monitoring period and which AOCs were potentially major PFCA sources in microenvironments. This market trend monitoring study provided a means to conduct independent assessment of the degree of success of the PFOA Stewardship Program. Priority was given to monitoring the market trends of the AOC categories established in Phase 1 that had the highest exposure potential, so samples with the highest PFCA content were selected. The results of the PFCA analyses were compared with the results acquired in Phase 1. In addition to seeking a general understanding of the market trends, this study also was intended to determine whether short-chain, fluorinated compounds are being used as alternatives and whether PFOS-related substances were still being used in AOCs.

2. Conclusions

To the best of the authors' knowledge, this is the first time that the temporal market trends of C4 to C12 PFCA contents in a wide variety of AOCs have been reported. In conclusion, large reduction of PFOA was observed in each of the AOC categories with the exception of one product in the home textile and upholstery category and two thread-sealant tape products, for which increased PFOA concentrations were detected. It was observed that the PFBA-C4 content increased in 19 of the 35 AOCs monitored, with all floor wax products showing significant increases in the amount of PFBA-C4. The latest monitoring data suggest that commercial carpet-care liquids, treated floor waxes, treated food contact paper, and thread-sealant tapes are likely the most significant sources of the nine PFCAs, including PFOA, among the 10 article categories that were studied. The data presented here indicate that the concentrations of PFCAs in the AOCs have decreased rapidly in recent years. The observed trends of the relative increase of PFBA-C4 and PFBS-C4S confirm that perfluorinated substances that have shorter chain are being used as alternatives to long-chain PFCAs. However, no significant difference in trends was observed for short-chain PFCA (sum of C4 to C7) versus long-chain PFCA (sum of C8 to C12) over the monitored period. Our limited data show that the PFOA Stewardship Program has produced the following measurable results: (1) the availability of consumer articles that have been treated with fluorinated chemicals is declining; (2) PFCA content has declined significantly for most of the AOCs, although the PFCA content of a few samples still remains high; and (3) PFOS is still being used in the market, and PFBS-C4 is being used as an alternative for PFOS for some products. Global collaboration is needed to further reduce the PFCA content in consumer articles. The results of this study can help inform risk management decisions on the changing market for PFCAs as well as provide insight for designing future research work.

3. Recommendations

This study demonstrated the importance of monitoring over time the use and variations in emergence of environmental pollutants in products and the marketplace. Also demonstrated was the efficacy of the measures taken to reduce the release and subsequent exposure associated with their use. However, the data presented here provide only a limited assessment of the usage trends for PFCAs in AOCs over time due to the limited number of samples, the relatively short duration of data collection, the inability to obtain the same products on the market, and the statistical uncertainties associated with limited quantities of data. Related research is recommended to: (1) extend the market monitoring period for the AOCs studied, including new products that are likely to contain PFCAs; (2) monitor perfluorinated telomeric substances and polyfluorochemicals in AOCs because some of them are known to be precursors of PFCAs; (3) monitor PFAS in the market; and (4) study the mechanisms that lead to the transfer of PFCAs from sources to indoor air, dust, and surfaces, especially during the use of consumer products.

4. Materials and Methods

4.1 Sample Collection

The first step of the long-term market monitoring effort was the purchase of AOCs between 2007 and 2011 for ten of the thirteen categories identified in Phase 1. Three product categories (i.e., non-stick cookware, dental floss, and miscellaneous) were not monitored due to low PFC content or low market availability. The ten AOC categories and the numbers of AOCs purchased for each category are presented in Table 4-1.

A total of 95 samples from 35 AOCs were collected and analyzed over the four-year period. Whenever possible, the exact product was purchased based on the product barcode information recorded for the original samples. This has proven to be a very limiting factor for the monitoring because of the ever-changing markets, especially for clothing (apparel and membranes) and carpet products. With the exception of school uniforms, exact duplicate products for apparel were impossible to purchase. The school uniforms retained the barcode identity, but some of the new products had different countries of origin. Carpets proved to be the most challenging. Carpets are manufactured in runs using the same dye and yarn formulations over the production life of a particular style. Some styles are in production for years, while others are replaced with newer fibers and manufacturing technologies more rapidly. The carpet-care solutions in both the commercial and household categories retained product continuity, as did the food contact products and non-woven medical garments. If it became necessary to replace products with similar products, the following three criteria were considered: (1) same manufacturer, (2) similar stain-resistant properties, and (3) similar formulations as indicated on the product label.

Table 4-1. AOCs analyzed for monitoring market trends

Category ID	Category name	Purchase Year			
		2007-2008 [a]	2009 [b]	2010	2011
A	Pre-treated carpet	3	0	4	2
B	Commercial carpet-care liquids	4	4	0	4
C	Household carpet/fabric-care liquids and foams	6	1	3	3
D	Treated apparel	5	4	0	6
E	Treated home textile and upholstery	2	0	2	2
F	Treated non-woven medical garments	3	3	0	3
G	Treated floor waxes and stone/wood sealants	4	0	0	4
H	Treated food contact paper	3	0	3	3
I	Membranes for apparel	3	0	3	2
J	Thread sealant tapes and pastes	2	0	2	2
Total		35	12	17	31

[a] Purchase dates were from 2007 to early 2008; [b] Purchase dates were from late 2008 to 2009.

4.2 **Sample Handling, Storage and Preparation**

The market-trend monitoring project followed specific methods for handling, storing, extracting, and analyzing the samples, as well as the QA measures and criteria that were established during Phase I [3].

AOCs were purchased from local retailers and online stores. Samples were kept in their original packaging and transported to the EPA laboratory, where they were photographed and the product information was logged into the AOC record notebook. Product name, vendor name, manufacturer, purchase date, price, quantity, information indicating possible use of fluorinated compounds, and other descriptive details were documented.

After being logged into the database, small samples of solid AOCs, such as fabrics or carpets, were cut from the primary sample in a set of ten 5 × 5 cm coupon samples and ten 10 × 10 cm samples using a 60-mm rotary cutter and scissors. The subdivided sets were triple wrapped in aluminum foil, placed in appropriately-labeled plastic bags and stored in a refrigerator. The rest of the primary sample and any additional pieces of the same AOC were wrapped in three layers of aluminum foil, placed in labeled plastic storage bags, and stored in an air-conditioned storage facility.

Multiple 5 × 5 cm subsample coupons were selected from each AOC to achieve the desired sample weight for extraction. The weight necessary for extraction and analysis was determined during Phase 1. Triplicate subsample sets were prepared for each AOC and placed in a desiccator overnight, after which, the samples were removed from the desiccator, weighed, and placed in labeled 50-mL polypropylene vials. Then the samples were transferred to the laboratory for extraction.

Liquid samples were logged into the database and then subdivided into 30-mL polyethylene vials. The vials were wrapped in three layers of aluminum foil, placed in labeled plastic storage bags, and stored in a refrigerator. The subsamples were transferred to the laboratory for extraction. The remaining primary liquid and any additional bottles of the same AOC were wrapped in three layers of aluminum foil and placed in labeled plastic storage bags.

4.3 **Sample Extraction and Analysis**

Sample coupons of solid AOCs were extracted with 45 mL of methanol and 100 µL of 2 ng/µL recovery check standard (RCS) in the 50-mL polyethylene centrifuge vials (BD Falcon™) by using a Nutating Mixer (Model VSN-5, PRO Scientific, Inc., CT, USA) at a speed setting of two for 24 ± 2 hours. After extraction, the methanol extract was transferred to a 170-mL borosilicate-glass concentration tube with stem (LabConco, MO, USA). The extraction vials were rinsed three times with approximately 3 mL of methanol. The glass concentration tubes were placed in a RapidVap N_2 Evaporation System (Model 791000, LabConco, MO, USA) that was custom-made without tetrafluoroethylene parts and coatings. The volume of the extract was reduced to approximately 1.5 mL, which were transferred into a 3-mL syringe with a 0.1 µm Anotop 25 filter attached and filtered into a clean 10-mL volumetric flask. The concentration tube was rinsed five times with approximately 1.5 mL of solution consisting of 60% (v/v)

methanol and 40% (v/v) 2 mN ammonium acetate aqueous solution (hereafter referred to as the 60:40 solution). Each 10-mL flask received 100 µL of 0.5 ng/µL internal standard (IS) and was brought to volume with the 60:40 solution. The volumetric flasks were placed in a sonication bath for 10 minutes, and the contents were transferred to a 15-mL polypropylene storage vial with appropriate label. The samples were stored at 4 °C in the refrigerator in the laboratory and were analyzed within 30 days.

For the extraction of liquid samples, a target amount of the samples (typically 1 g) was weighed and transferred from the 30-mL polyethylene storage vial to a 25-mL volumetric flask with 100 µL of 2 ng/µL RCS and brought to volume with the 60:40 solution. Then the flasks were sonicated for 10 minutes. After vacuum filtration via a 0.22-µm of Corning filter, 9.9 mL of the filtrate were transferred to a 10-mL volumetric flask with 100 µL of the 0.5 ng/µL internal standard. The Corning filter and flask were not rinsed during this time. It was necessary to filter some of the liquid samples again through a 0.1-µm Anotop filter by syringe (Micro-Mate®, Sigma-Aldrich). Then, each flask was sonicated for 10 minutes. The sample contents were transferred to a 15-mL polypropylene storage vial, after which the vial was labeled appropriately and stored at 4°C in a refrigerator. The samples were analyzed within 30 days.

Sample quantification was conducted using an Agilent 1100 HPLC equipped with an Applied Biosystem API 3200 Triple Quadrupole Mass Spectrometer (LC/MS/MS) with a Turbo V ion-spray interface. The analytes included nine PFCAs (C4 to C12) and five sulfonates, which are listed in Table 4-2. The PFCAs and sulfonates were calibrated separately. An isotopically-labeled PFCA (perfluoro-n-[1, 2-$^{13}C_2$] decanoic acid) was used as the extraction recovery check standard for PFOA and its homologues. An isotopically-labeled sulfonate standard (sodium perfluoro-1-hexane [$^{18}O_2$] sulfonate) was used for PFOS and its homologues. The internal standards were perfluoro-n-[1, 2, 3, 4-$^{13}C_4$] octanoic acid for PFCAs and sodium perfluoro-1-[1, 2, 3, 4-$^{13}C_4$] octanesulfonate for PFAS analysis. Each batch was analyzed along with its corresponding QC samples. The conditions of the instrument are presented in Table 4-3 and Table 4-4.

Table 4-2. Analyte names, chemical formulas, and chemical abstracts service registration numbers

Analyte name	Short name	Chemical formula	CAS#
perfluorobutyric acid	PFBA-C4	$C_4HF_7O_2$	375-22-4
perfluoropentanoic acid	PFPeA-C5	$C5HF_9O_2$	2706-90-3
perfluorohexanoic acid	PFHxA-C6	$C_6HF_{11}O_2$	307-24-4
perfluoroheptanoic acid	PFHpA-C7	$C_7HF_{13}O_2$	375-85-9
perfluorooctanoic acid	PFOA-C8	$C_8HF_{15}O_2$	335-67-1
perfluorononanoic acid	PFNA-C9	$C_9HF1_7O_2$	375-95-1
perfluorodecanoic acid	PFDA-C10	$C_{10}HF_{19}O_2$	335-76-2
perfluoroundecanoic acid	PFUnDA-C11	$C_{11}HF_{21}O_2$	2058-94-8
perfluorododecanoic acid	PFDoDA-C12	$C_{12}HF_{23}O_2$	307-55-1
perfluoro-n-[1,2,3,4-$^{13}C_4$] octanoic acid (IS)	PFOA-C8-$^{13}C_4$	$^{13}C_4{}^{12}C_4HF_{15}O_2$	--
perfluoro-n-[1,2-^{13}C2] decanoic acid (RCS)	PFDA-C10-$^{13}C_2$	$^{13}C_2{}^{12}C_8HF_{15}O_2$	--
potassium perfluoro-1-butanesulfonate	PFBS-C4	$C_4F_9SO_3K$	29420-49-3
sodium perfluoro-1-hexanesulfonate	PFHxS-C6	$C_6F_{13}SO_3Na$	108427-53-8
sodium perfluoro-1-heptanesulfonate	PFHpS-C7	$C_7F_{15}SO_3Na$	--
sodium perfluoro-1-octanesulfonate	PFOS-C8	$C_8F_{17}SO_3Na$	1763-23-1
sodium perfluoro-1-decanesulfonate	PFDS-C10	$C_{10}F_{21}SO_3Na$	--
sodium perfluoro-1-[1,2,3,4-$^{13}C_4$]octanesulfonate (IS)	PFOS-C8-$^{13}C_4$	$^{13}C_4{}^{12}C_4F_{17}SO_3Na$	--
sodium perfluoro-1-hexane[$^{18}O_2$]sulfonate (RCS)	PFHxS-C6-$^{18}O_2$	$C_6F_{13}S^{18}O_2{}^{16}ONa$	--

Table 4-3. Operating conditions for the Agilent 1100 HPLC/Applied Biosystem API 3200 triple quadrupole mass spectrometer for the analysis of PFCAs and PFAS

LC Parameters	**Settings**			
Column	Agilent Zorbax Eclipse XDB-C18, 2.1 x 50 mm, 3.5 µm			
Guard column	Agilent Eclipse XDB-C18, 2.1 x 15 mm, 3.5 µm			
Oven temperature	50 ºC			
Injection volume	20 µL			
Mobile phase	2 mM ammonium acetate -H_2O (A), MeOH (B)			
Flow rate	250 µL/min			
Gradient	Steps	Time (min)	%A	%B
	0	4	35	65
	1	5	20	80
	2	7	20	80
	3	12	10	90
	4	12.5	35	65
Mass Spectrometric Parameters				
Ion source	Turbo spray			
Curtain gas (CUR)	10 arbitrary unit (setting)			
Collision gas (CAD)	3 arbitrary unit (setting)			
Ion spray voltage (IS)	- 4500 V			
Temperature (TEM)	425 °C			
Ion source gas (GS1)	14 arbitrary unit (setting)			
Ion source gas (GS2)	4 arbitrary unit (setting)			
Interface heater (ihe)	On			
Scan type	MRM			
Polarity	Negative			
Resolution Q1	Unit			
Resolution Q2	Unit			

Table 4-4. Analyte-dependent mass spectra parameters

Analyte	Q1 Mass (amu)	Q3 Mass (amu)	Time (ms)	DP (V)	EP (V)	CE (V)	CXP (V)
PFC-Acids							
PFBA-C4	213	169	125	-20	-4.5	-12	-4
PFPeA-C5	263	219	125	-10	-3	-12	-4
PFHxA-C6	313	269	125	-15	-4	-12	-4
PFHpA-C7	363	319	125	-15	-4.5	-12	-6
PFOA-C8	413	369	125	-15	-4.5	-14	-6
PFOA-C8-$^{13}C_4$	417	372	125	-15	-4.5	-14	-6
PFNA-C9	463	419	125	-15	-5	-12	-6
PFDA-C10	513	469	125	-12	-4.5	-8	-8
PFDA-C10-$^{13}C_2$	515	470	125	-10	-6	-10	-38
PFUnDA-C11	563	519	125	-15	-7	-6	-8
PFDoDA-C12	613	569	125	-10	-9	-18	-10
PFC-Sulfonates							
PFBS-C4	299	99	125	-45	-5.5	-38	-2
PFHxS-C6	399	99	125	-55	-9.5	-52	-2
PFHxS-C6-$^{18}O_2$	403	103	125	-55	-9	-52	0
PFHpS-C7	449	99	125	-50	-7	-85	-9
PFOS-C8	499	99	125	-75	-8	-66	-2
PFOS-C8-$^{13}C_4$	503	99	125	-70	-6	-118	-56
PFDS-C10	599	99	125	-80	-12	-74	-2

4.4 **Quality Assurance and Quality Control**

Quality assurance (QA) and quality control (QC) procedures were implemented in this project by following the guidelines and procedures detailed in the approved Category II Quality Assurance Project Plan (QAPP).

4.4.1 Data Quality Indicators Goals

The QA measures and criteria remained the same as those for Phase 1. Data quality indicator (DQI) goals for the measurement parameters and validation methods are listed in Table 4-5.

Table 4-5. Data quality indicator goals for critical measurements

Measurement	Parameters	Objective	Method
Quantification by the LC/MS/MS method	Instrument detection limit	≤0.2 ng/mL	EPA method (40 CFR 136, 1986) [14]
	Accuracy	85-115%	DCC and IAP
	Precision	±20%	RSD for replicate or triplicate injections
	Calibration	0.99	Coefficient of determination (r^2) of linear regression
Solvent extraction	Accuracy	80-120%	Recovery check standard
	Precision	±20%	RSD for replicate or triplicate extractions
	System blank	< MDL	Extraction without AOC sample
Weight of AOC samples	Accuracy	±2 mg	NIST-traceable weights

4.4.2 Instrument Calibration

The LC/MS/MS was calibrated at eight concentration levels with triplicate injections in the concentration range of 0.3 to 160 ng/mL for PFCAs and 0.3 to 100 ng/mL for PFAS. The instrument was recalibrated when the QC samples were off the acceptable range. The Internal Audit Program (IAP) was instituted to assess the accuracy and precision of the LC/MS/MS system. IAP standards were prepared by someone other than the person who prepared the calibration standards by using at least three PFCAs or PFAS, including PFOA or PFOS, obtained from a second source. The IAP standards without stating their concentrations were submitted to the analyst as a measure for calibration verification.

The balance was calibrated once a year by experts in the EPA Metrology Laboratory using NIST traceable weights. Before and after a subsample was weighed, the balance was checked with NIST-traceable weights, and the error was within 2 mg.

4.4.3 Detection Limit

The method detection limit (MDL) was not investigated for the project. After each calibration, the instrument detection limit (IDL) was determined by analyzing the lowest calibration standard seven times and then calculating three standard deviations from the measured concentrations of the standard.

4.4.4 Quality Control Samples

Quality control samples consisted of field blanks, solvent blanks, duplicates or triplicates, daily calibration checks and recovery check standards.

With each daily batch of extractions, a set of five QC samples was prepared and analyzed. Each set consisted of one field blank, one solvent blank, and three blanks for the recovery check standards. The field blank was taken through the entire extraction process similar to the corresponding AOC samples. The solvent blank was prepared in a clean, 10-mL flask with 9.9 mL of 60:40 solution and 100 µL of the internal standard. The three RCS blanks were prepared with 9.8 mL of 60:40 solution, 100 µL of IS, and 100 µL of RCS in a 10-mL flask. These five flasks were sonicated for 10 minutes and transferred to labeled polypropylene vials. The QC samples were stored in the refrigerator with each batch of samples to be analyzed. Samples were analyzed in batches, including AOC samples in duplicate or triplicate, quality control samples, and two daily calibration check standards analyzed at the beginning and the end of the analysis sequence.

5. Results

5.1 **Distribution of AOC Samples**

Ninety-five samples from 35 types of AOCs were obtained between 2007 and 2011 (Table 4-1). The dates the samples were manufactured were unknown, because the product labels did not include that information. The purchase data and sample descriptions are presented in Tables 5-1 through 5-10. The AOC ID numbers were kept consistent with those used in Phase 1. For example, A-1-0 refers to the AOC in category A, product 1, and the first purchase, which was measured during Phase 1 of the project. A-1-1 is the same AOC in category A, product 1, second purchase. No AOC samples were collected under categories of cookware, dental floss, and miscellaneous.

Table 5-1. AOC samples collected for pre-treated carpeting

AOC ID	Purchase Date	Description	Country of Origin
A-1-0	03/09/2007	Nylon carpet 1	USA
A-1-1	05/18/2010	Nylon carpet 2	USA
A-1-2[a]	09/08/2011	Nylon carpet 3	USA
A-1-3 [a]	09/08/2011	Nylon carpet 4	USA
A-2-0	03/12/2007	Corn polymer carpet 1	USA
A-2-1 [a]	05/18/2010	Corn polymer carpet 2	USA
A-2-2 [a]	05/18/2010	Corn polymer carpet 3	USA
A-9-0	02/4/2008	Polypropylene carpet 1	USA
A-9-1	05/18/2010	Polypropylene carpet 2	USA

[a] The AOCs were not exactly the same products even though they were purchased on the same day.

Table 5-2. AOC samples collected for commercial carpet/fabric-care liquids

AOC ID	Purchase Date	Description	Country of Origin
B-1-0	04/19/2007	Carpet/upholstery protector concentrate 1	USA
B-1-1	05/26/2009	Carpet/upholstery protector concentrate 1	USA
B-1-2	02/23/2011	Carpet/upholstery protector concentrate 1	USA
B-3-0	04/19/2007	Solvent-based fabric protector	USA
B-3-1	11/24/2008	Solvent-based fabric protector	USA
B-3-2	02/23/2011	Solvent-based fabric protector	USA
B-5-0	04/19/2007	Carpet/upholstery protector concentrate 2	USA
B-5-1	11/24/2008	Carpet/upholstery protector concentrate 2	USA
B-5-2	02/23/2011	Carpet/upholstery protector concentrate 2	USA
B-7-0	04/19/2007	Ready-to-use carpet protector 1	USA
B-7-1	05/1/2008	Ready-to-use carpet protector 1	USA
B-7-2	02/23/2011	Ready-to-use carpet protector 1	USA

Table 5-3. AOC samples collected for household carpet/fabric-care liquids and foams

AOC ID	Purchase Date	Description	Country of Origin
C-1-0	04/19/2007	Carpet Shampoo 1	USA
C-1-1	11/06/2008	Carpet Shampoo 1	USA
C-1-2	03/28/2011	Carpet Shampoo 1	USA
C-2-0	05/10/2007	Household carpet care 1	USA
C-2-1	02/23/2011	Household carpet care 1	USA
C-4-0	05/16/2007	Household carpet protector 1	USA
C-4-1	02/22/2010	Household carpet protector 1	USA
C-5-0	05/16/2007	Household carpet protector 2	USA
C-5-1	02/23/2011	Household carpet protector 2	USA
C-8-0	06/06/2007	Household carpet care 2	USA
C-8-1	03/26/2010	Household carpet care 2	USA
C-9-0	09/29/2007	Membrane fabric care 1	England
C-9-1	03/01/2010	Membrane fabric care 1	England

Table 5-4. AOC samples collected for treated apparel

AOC ID	Purchase Date	Description	Country of Origin
D-3-0	05/10/2007	Girl's uniform shirt	Vietnam
D-3-1	11/12/2008	Girl's uniform shirt	Vietnam
D-3-2	02/24/2011	Girl's uniform shirt	China
D-3-3	08/16/2011	Girl's uniform shirt	China
D-4-0	05/10/2007	Boy's uniform/dress shirt	Thailand
D-4-1	11/24/2008	Boy's uniform/dress shirt	El Salvador
D-4-2	02/24/2011	Boy's uniform/dress shirt	El Salvador
D-4-3	08/16/2011	Boy's uniform/dress shirt	Bangladesh
D-5-0	05/10/2007	Boy's uniform/dress pant	Dominican Republic
D-5-1	08/16/2011	Boy's uniform/dress pant	Dominican Republic
D-5-2[a]	02/24/2011	Boy's uniform pant	Dominican Republic
D-7-0	05/10/2007	Girl's uniform pant	China
D-7-1	11/12/2008	Girl's uniform pant	Vietnam
D-7-2	08/16/2011	Girl's uniform pant	China
D-10-0	08/17/2007	Man's dress pant	Malaysia
D-10-1	11/24/2008	Man's dress pant	Malaysia

[a] For PFAS only.

Table 5-5. AOC samples collected for treated home textile and upholstery

AOC ID	Purchase Date	Description	Country of Origin
E-7-0	07/10/2007	Mattress pad 1	USA
E-7-1	02/22/2010	Mattress pad 1	USA
E-7-2	03/30/2011	Mattress pad 1	USA
E-8-0	07/10/2009	Mattress pad 2	USA
E-8-1	03/30/2010	Mattress pad 2	USA
E-8-2	03/30/2011	Mattress pad 2	USA

Table 5-6. AOC samples collected for treated non-woven medical garments

AOC ID	Purchase Date	Description	Country of Origin
F-2-0	01/30/2008	Surgical gown 1	Assembled in China with U.S. materials
F-2-1	05/05/2009	Surgical gown 1	China
F-2-2	03/28/2011	Surgical gown 1	China
F-3-0	01/30/2008	Surgical gown 2	China
F-3-1	05/05/2009	Surgical gown 2	China
F-3-2	03/28/2011	Surgical gown 2	China
F-4-0	01/30/2008	Surgical gown 3	Assembled in China with U.S. materials
F-4-1	05/05/2009	Surgical gown 3	China
F-4-2	03/28/2011	Surgical gown 3	China

Table 5-7. AOC samples collected for treated floor waxes and stone/wood sealants

AOC ID	Purchase Date	Description	Country of Origin
G-1-0	07/10/2007	Household floor wax 1	USA
G-1-1	02/23/2011	Household floor wax 1	USA
G-1-2	02/23/2011	Household floor wax 1	USA
G-2-0	07/10/2007	Household floor wax 2	USA
G-2-1	03/30/2011	Household floor wax 2	USA
G-4-0	07/10/2007	Commercial floor wax 1	USA
G-4-1	03/31/2011	Commercial floor wax 1	USA
G-6-0	07/10/2007	Commercial floor wax 2	USA
G-6-1	03/31/2011	Commercial floor wax 2	USA

Table 5-8. AOC samples collected for treated food contact paper

AOC ID	Purchase Date	Description	Country of Origin
H-3-0	10/15/2007	Food paper 1	USA
H-3-1	02/22/2010	Food paper 1	USA
H-3-2	09/07/2011	Food paper 1	USA
H-4-0	10/15/2007	Food paper 2	USA
H-4-1	02/22/2010	Food paper 2	USA
H-4-2	09/07/2011	Food paper 2	USA
H-5-0	10/30/2007	Food paper 3	USA
H-5-1	02/22/2010	Food paper 3	USA
H-5-2	09/12/2011	Food paper 3	USA

Table 5-9. AOC samples collected for membranes for apparel

AOC ID	Purchase Date	Description	Country of Origin
I-1-0	05/16/2007	Membrane 1	China
I-1-2	03/30/2010	Membrane 1	Vietnam
I-5-0	05/16/2007	Membrane 2	China
I-5-1	03/30/2010	Membrane 2	China
I-5-2	03/31/2011	Membrane 2	Indonesia
I-8-0	08/17/2007	Membrane 3	China
I-8-1	03/30/2010	Membrane 3	China
I-8-2	03/31/2011	Membrane 3	China

Table 5-10. AOC samples collected for thread-sealant tapes and pastes

AOC ID	Purchase Date	Description	Country of Origin
J-1-0	04/06/2007	Thread-sealant tape 1	Malaysia
J-1-1	03/28/2010	Thread-sealant tape 1	China
J-1-2	03/31/2011	Thread-sealant tape 1	China
J-6-0	08/17/2007	Thread-sealant tape 2	China
J-6-1	03/28/2010	Thread-sealant tape 2	China
J-6-2	03/31/2011	Thread-sealant tape 2	China

5.2 **Extractable PFCA Content in AOC Samples**

Figures 5-1 through 5-34 present PFCA contents detected in AOC samples. The data do not include values that were below the lowest calibration concentration or the value that did not meet QA criteria. All PFCA concentrations in one of the AOCs in the household carpet-care category, C-2, were below the lowest calibration concentration. Therefore, the data are not presented in the figures. The complete data of PFCA concentrations, including percentage of recovery check standards (%RCS) and the number of samples analyzed for each of the AOCs (N) are summarized in the Appendix A (Tables A-1 through A-10).

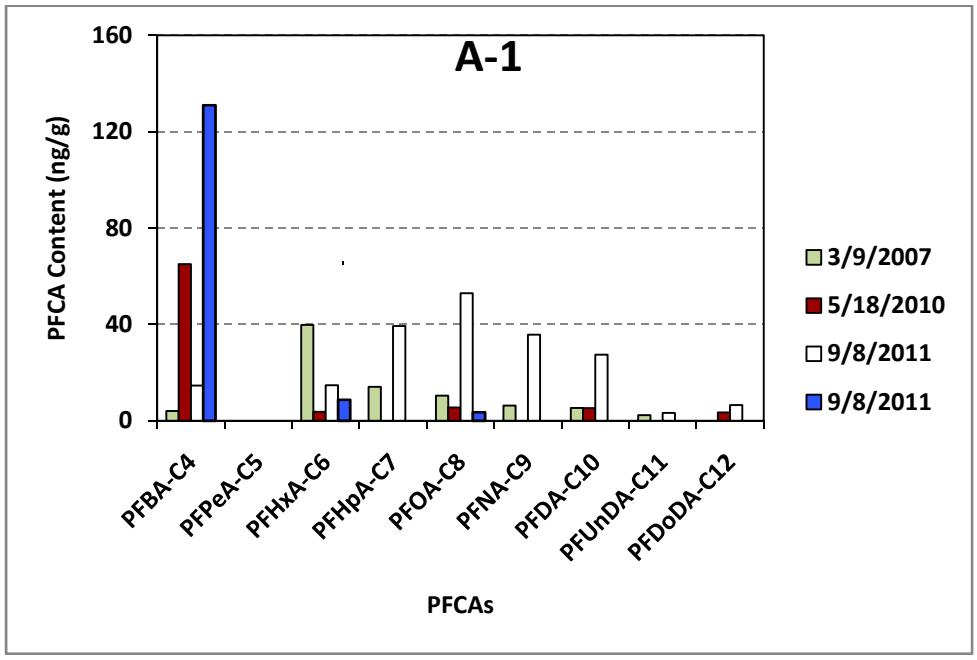

Figure 5-1. Trends of PFCAs detected in nylon carpet 1 (A-1) (Two slightly different products were purchased on 9/8/2011)

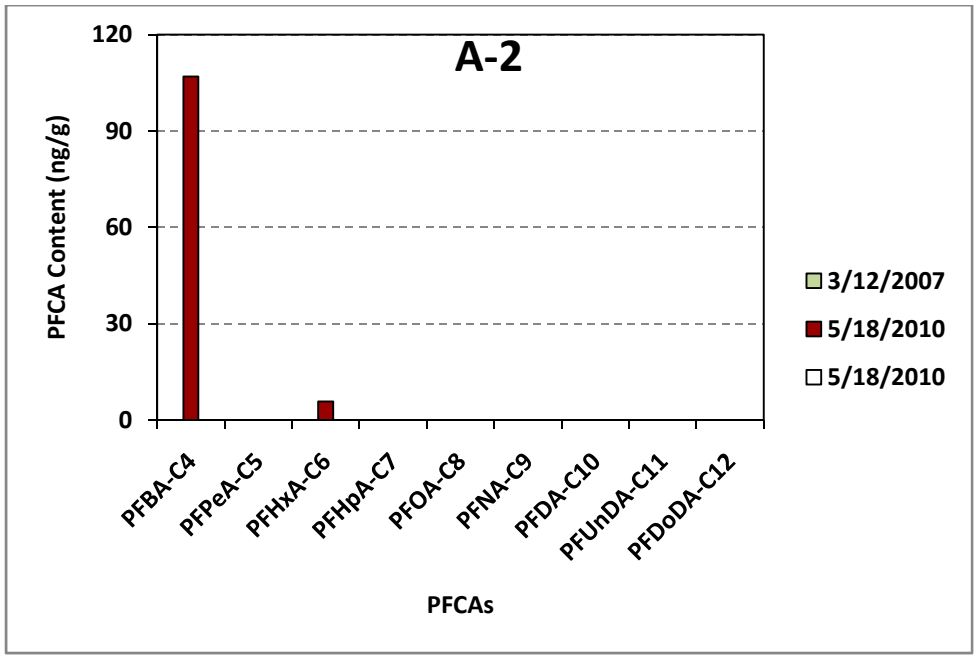

Figure 5-2. Trends of PFCAs detected in corn polymer carpet (A-2)

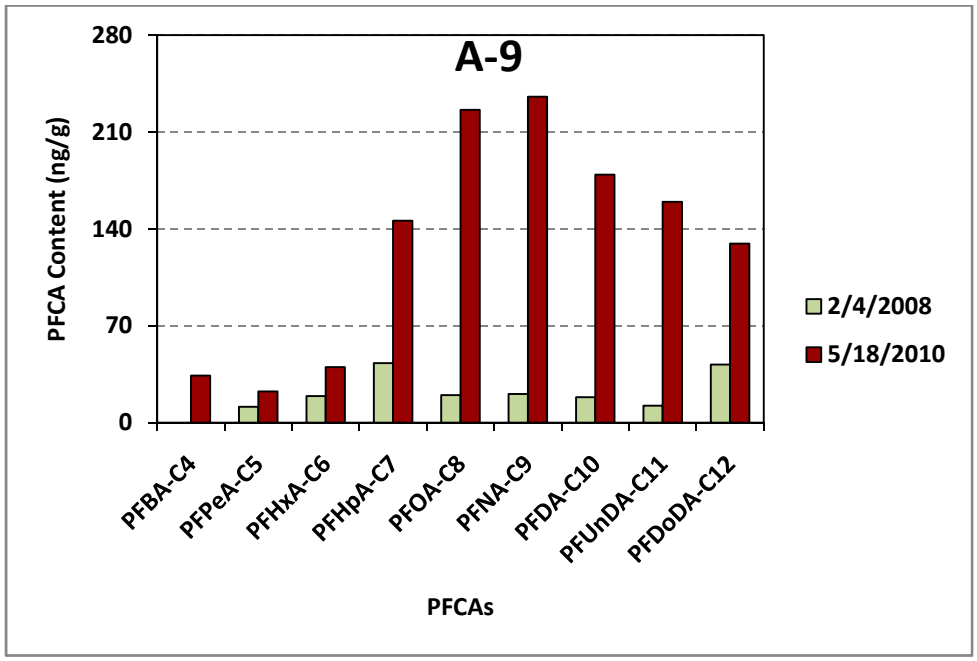

Figure 5-3. Trends of PFCAs detected in polypropylene carpet 4 (A-9)

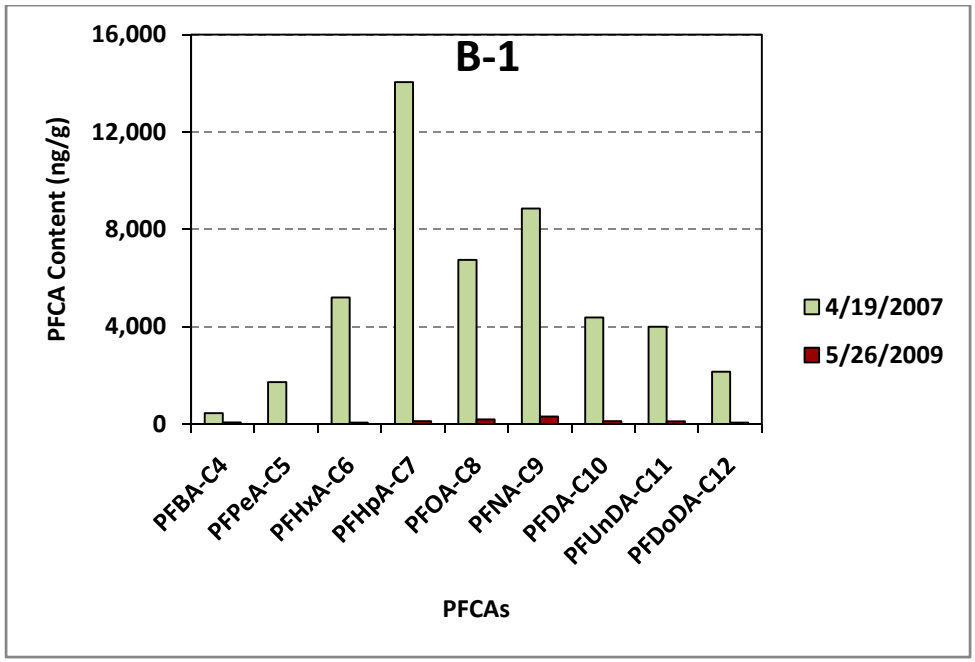

Figure 5-4. Trends of PFCAs detected in carpet/upholstery protector concentrate 1 (B-1)

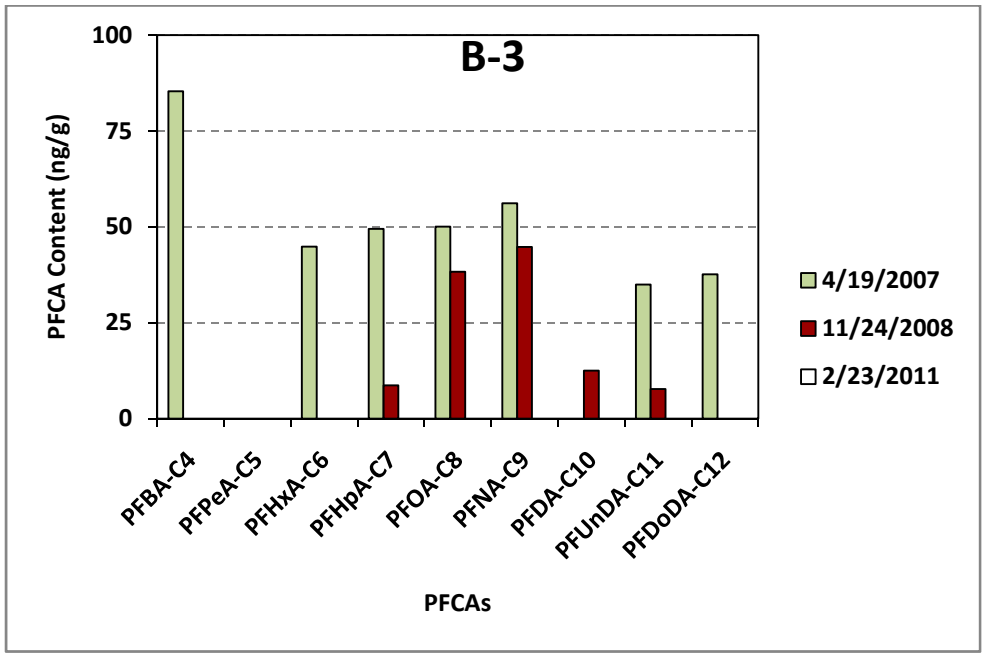

Figure 5-5. Trends of PFCAs detected in solvent based fabric protector (B-3)

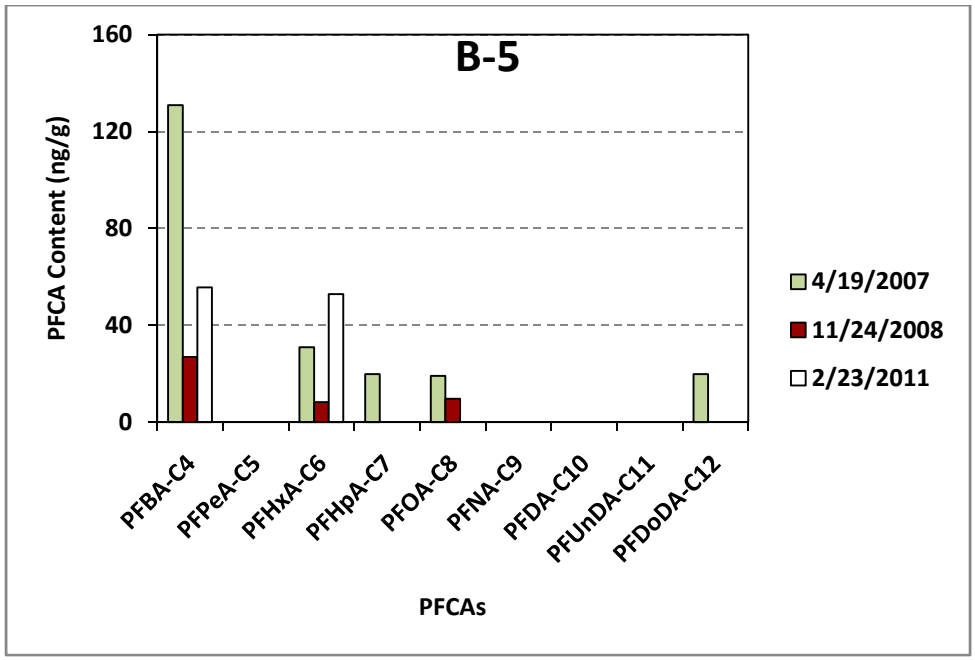

Figure 5-6. Trends of PFCAs detected in carpet/upholstery protector concentrate 2 (B-5)

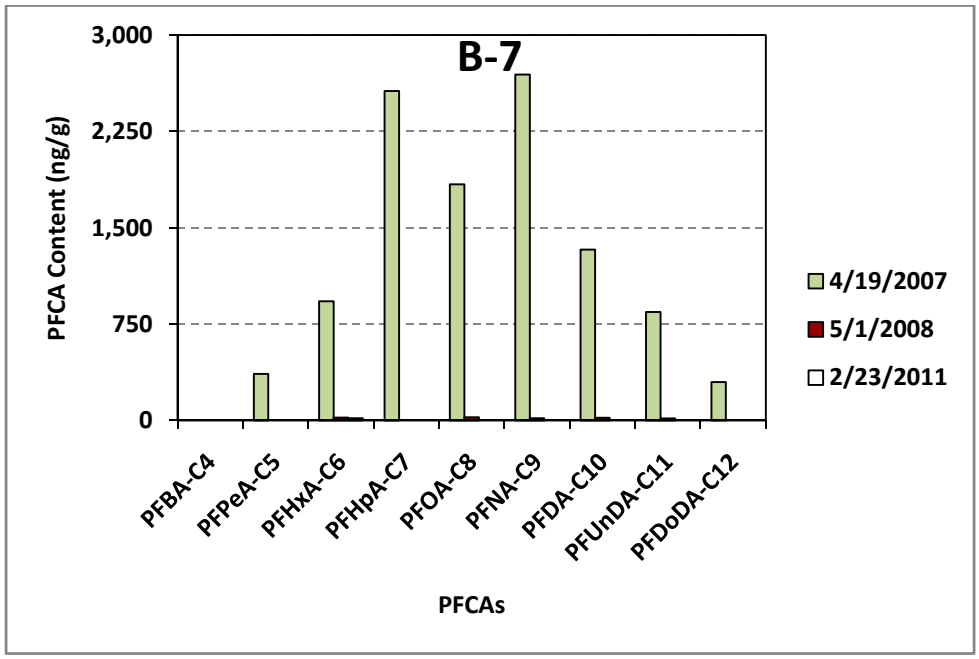

Figure 5-7. Trends of PFCAs detected in ready-to-use carpet protector 1 (B-7)

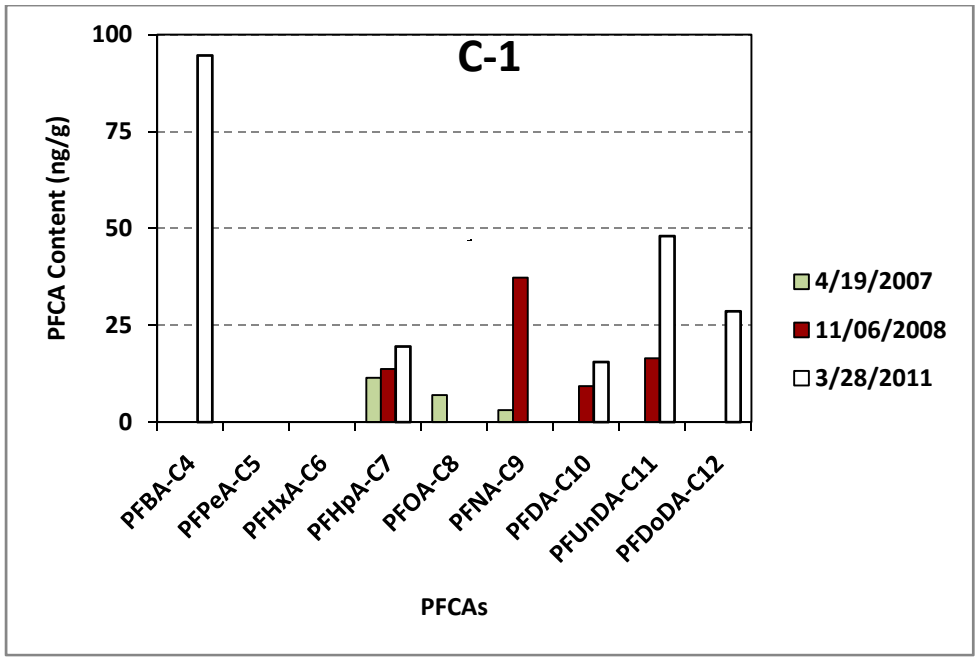

Figure 5-8. Trends of PFCAs detected in carpet shampoo 1 (C-1)

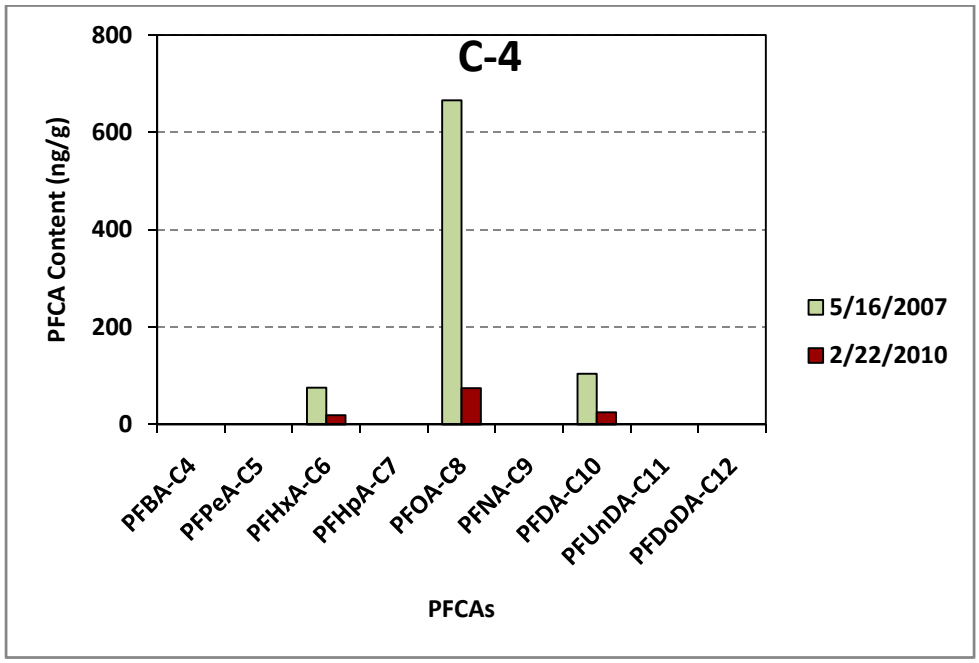

Figure 5-9. Trends of PFCAs detected in household carpet protector 1 (C-4)

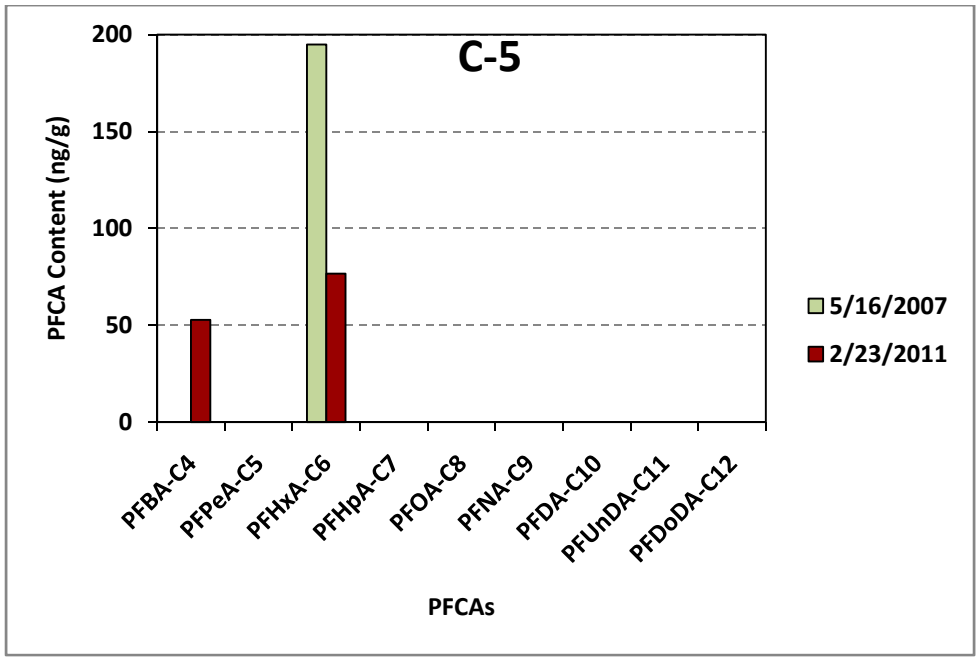

Figure 5-10. Trends of PFCAs detected in household carpet protector 2 (C-5)

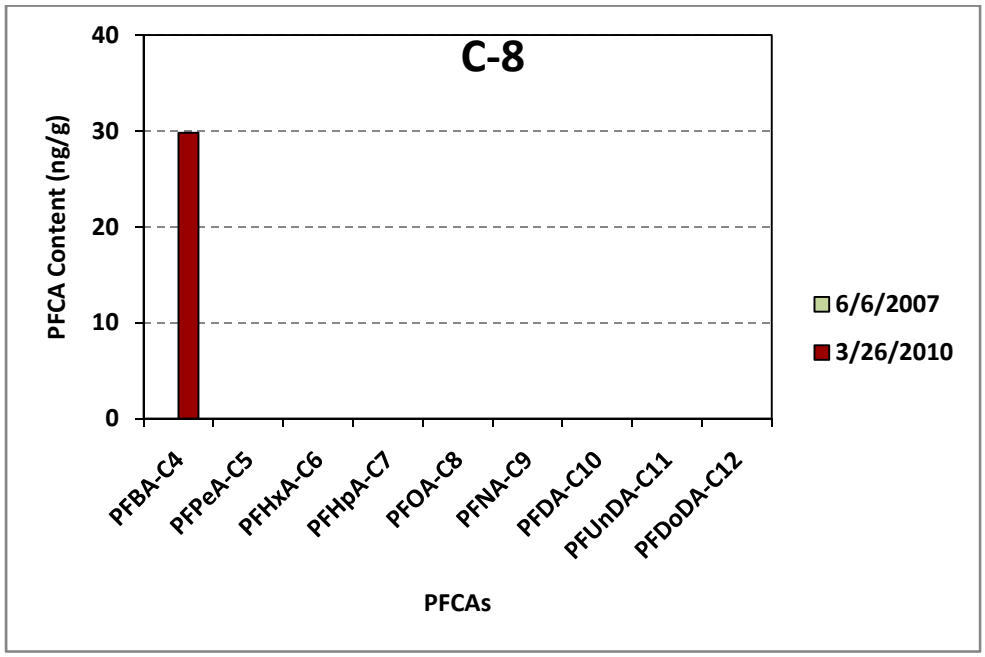

Figure 5-11. Trends of PFCAs detected in household carpet care 2 (C-8)

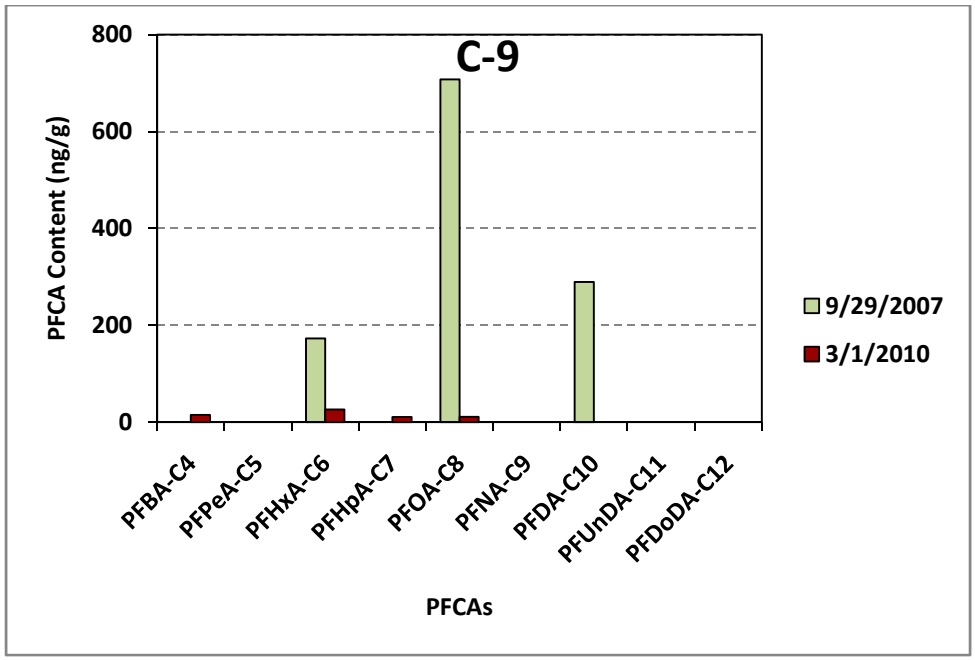

Figure 5-12. Trends of PFCAs detected in membrane fabric care 1 (C-9)

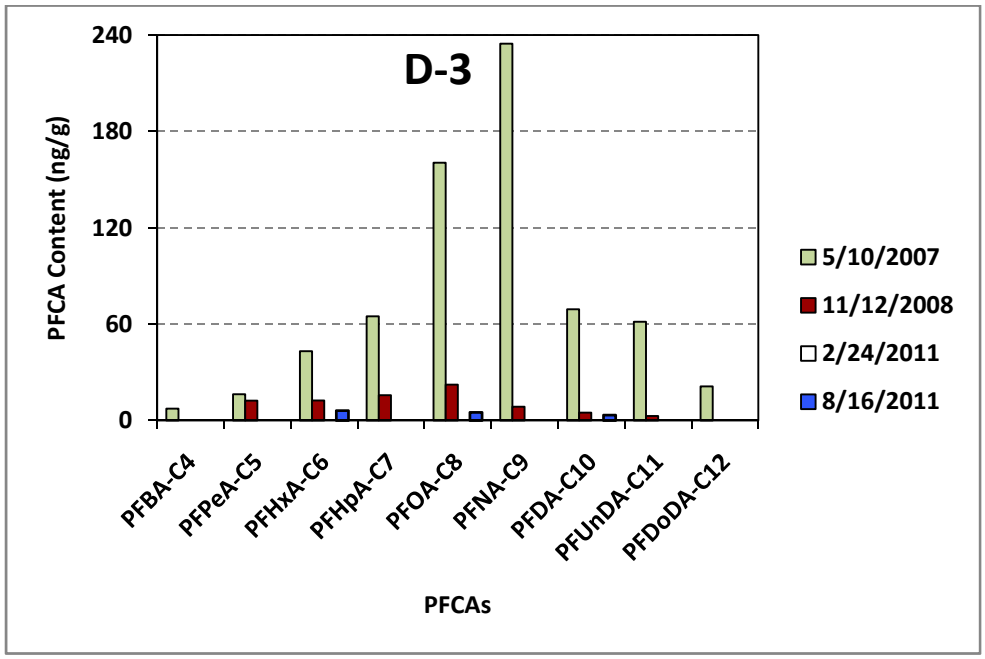

Figure 5-13. Trends of PFCAs detected in girl's uniform shirt (D-3)

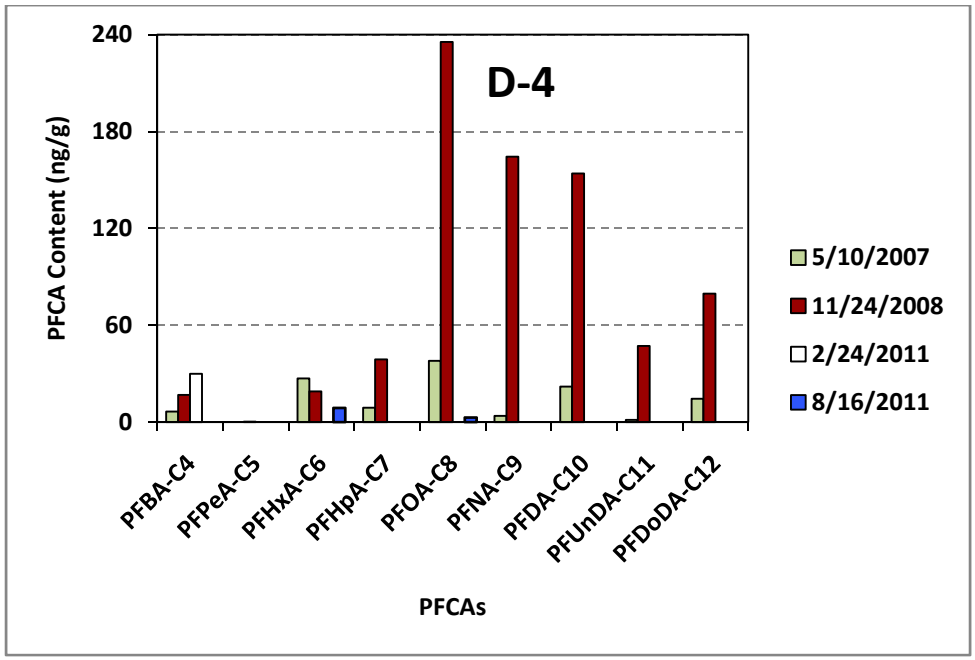

Figure 5-14. Trends of PFCAs detected in boy's uniform/dress shirt (D-4)

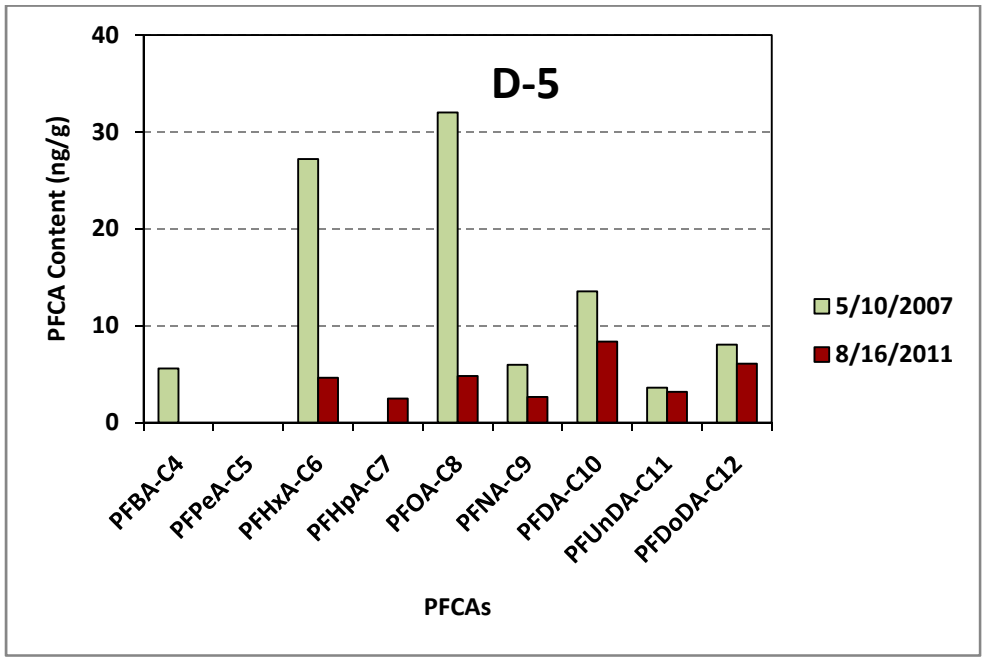

Figure 5-15. Trends of PFCAs detected in boy's uniform/dress pant (D-5)

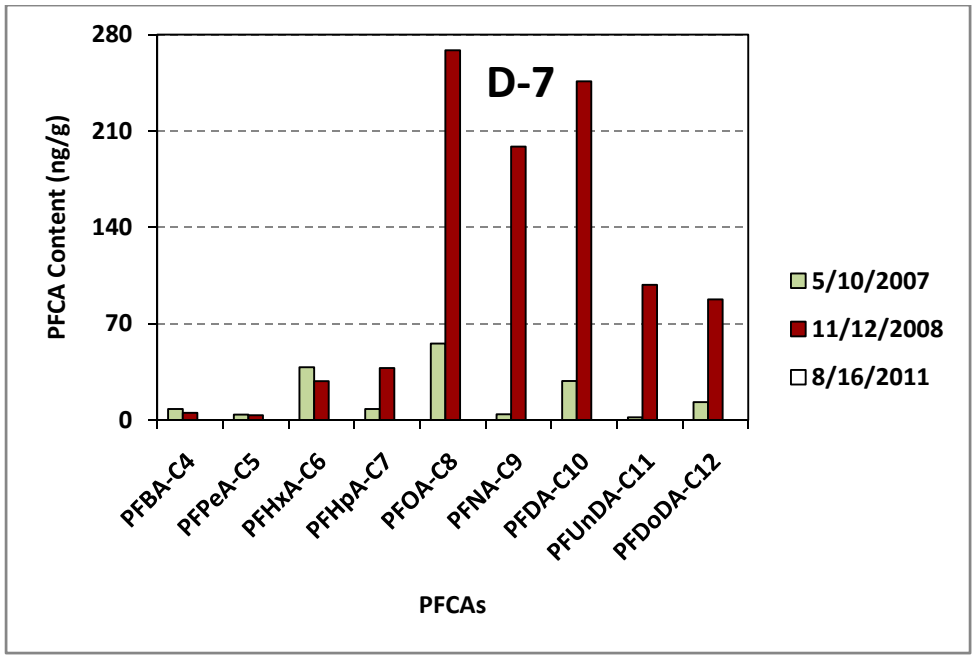

Figure 5-16. Trends of PFCAs detected in girl's uniform pant (D-7)

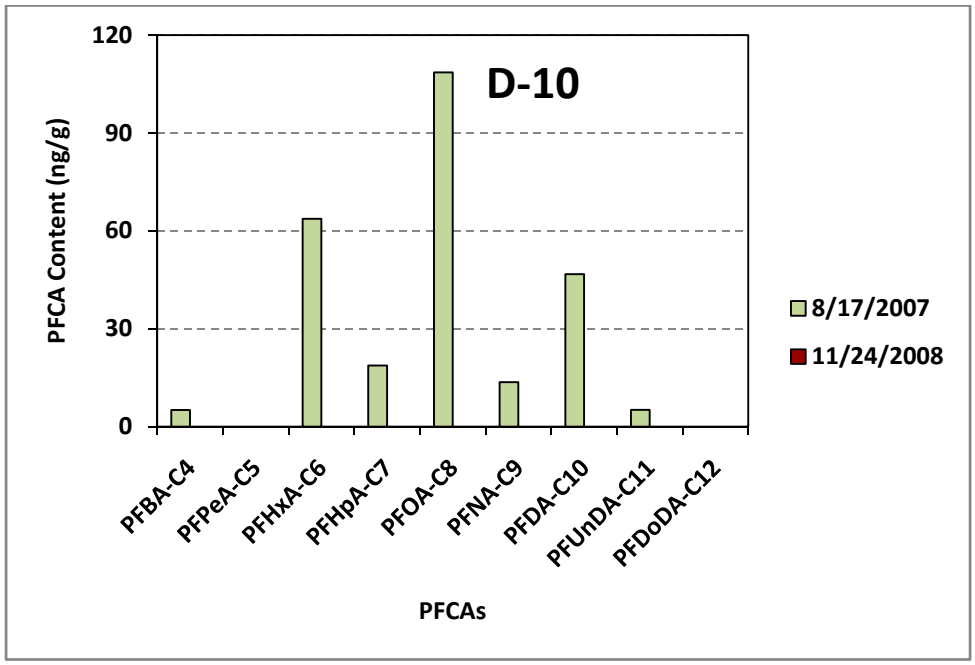

Figure 5-17. Trends of PFCAs detected in man's dress pant (D-10)

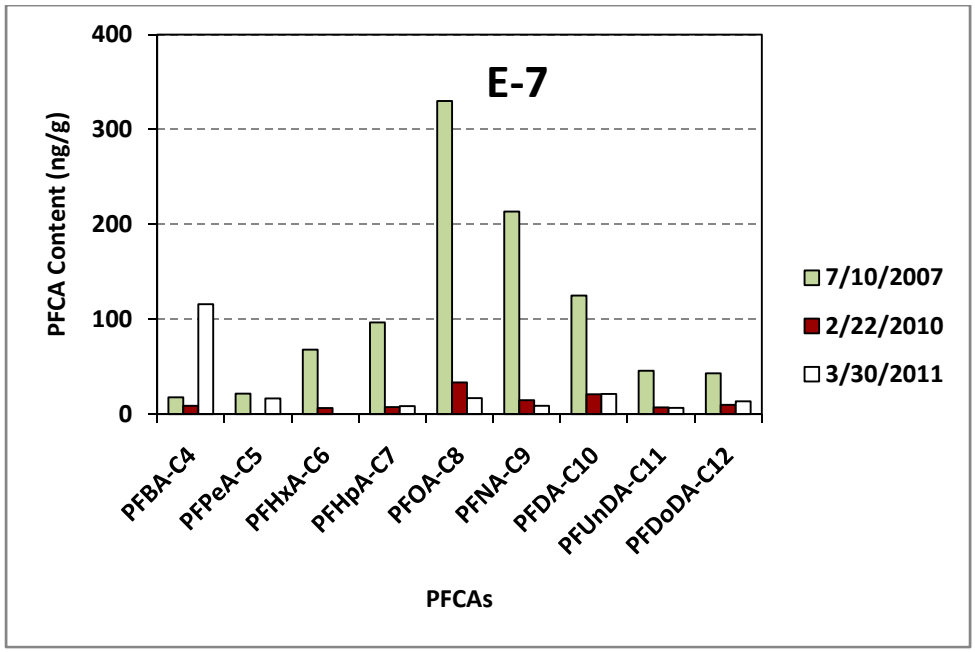

Figure 5-18. Trends of PFCAs detected in mattress pad 1 (E-7)

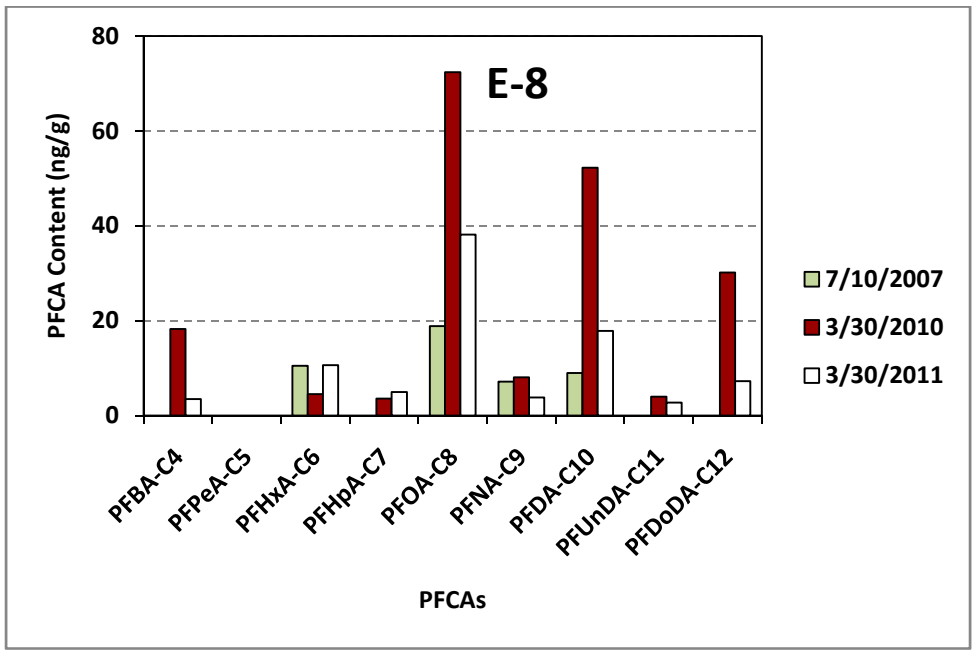

Figure 5-19. Trends of PFCAs detected in mattress pad 2 (E-8)

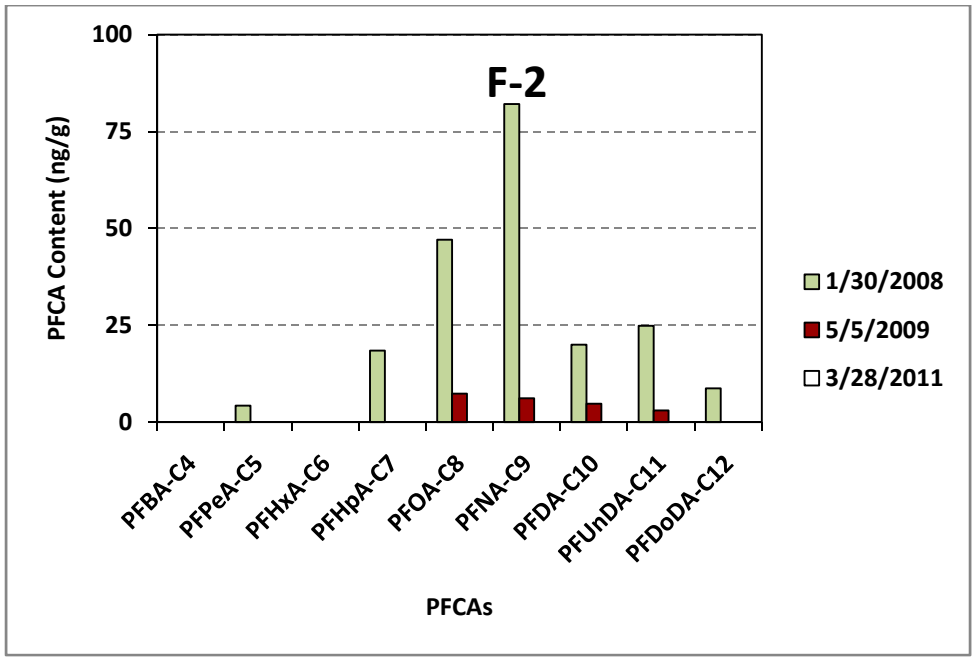

Figure 5-20. Trends of PFCAs detected in surgical gown 1 (F-2)

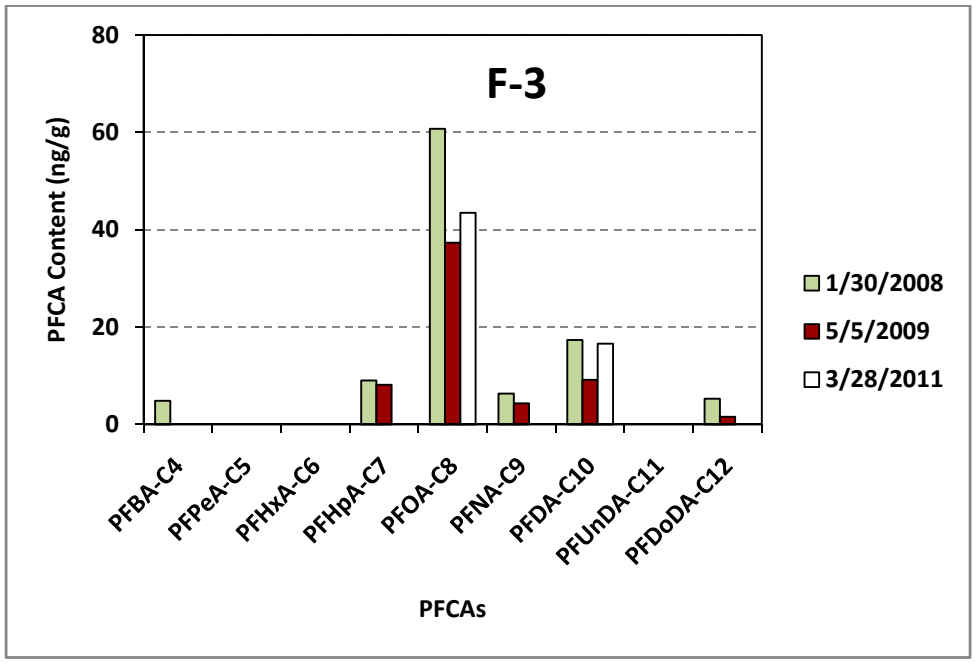

Figure 5-21. Trends of PFCAs detected in surgical gown 2 (F-3)

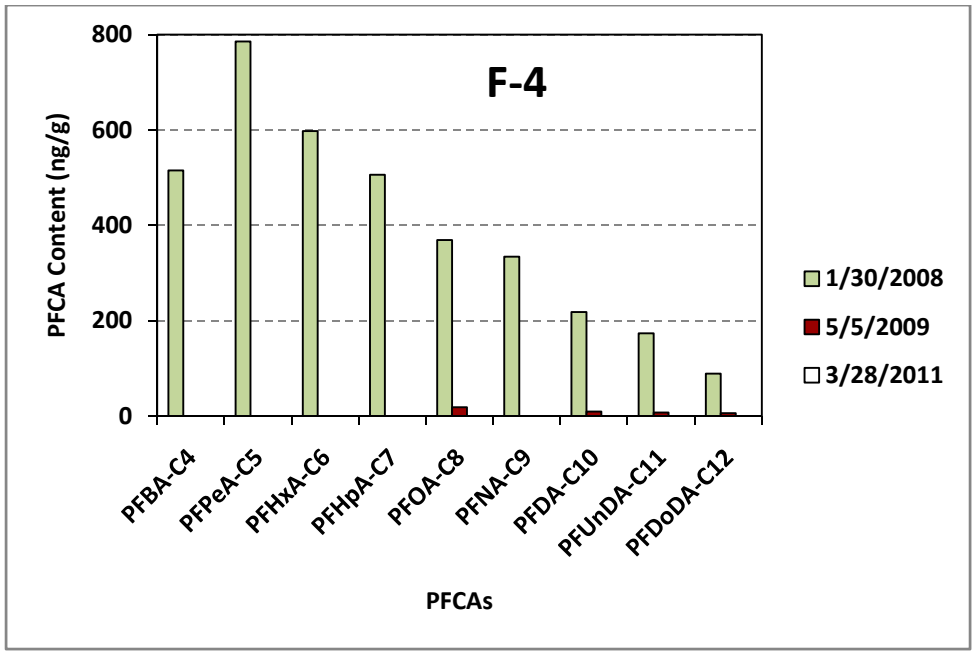

Figure 5-22. Trends of PFCAs detected in surgical gown 3 (F-4)

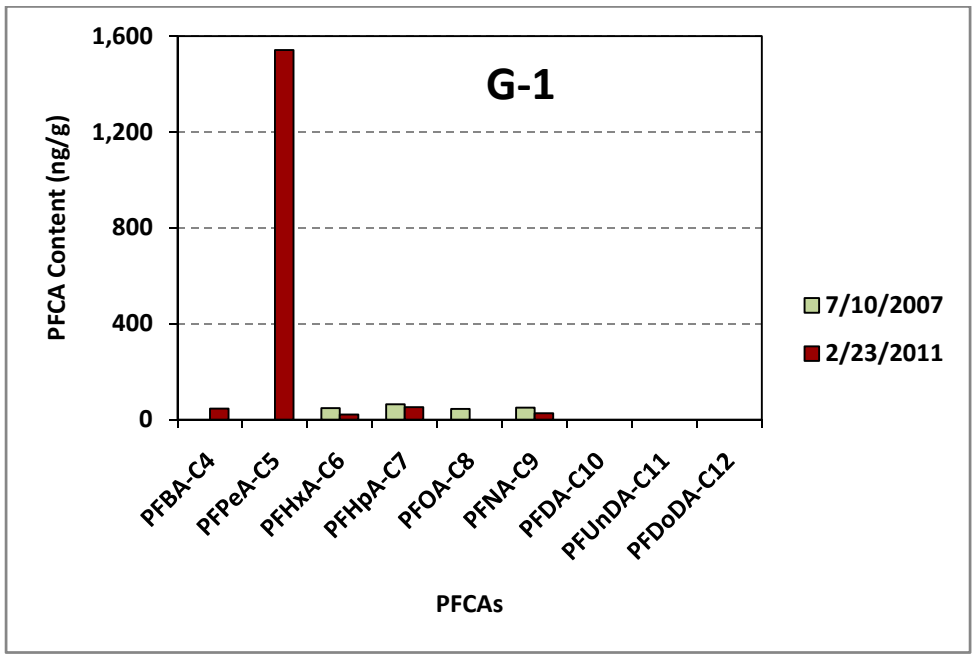

Figure 5-23. Trends of PFCAs detected in household floor wax 1 (G-1)

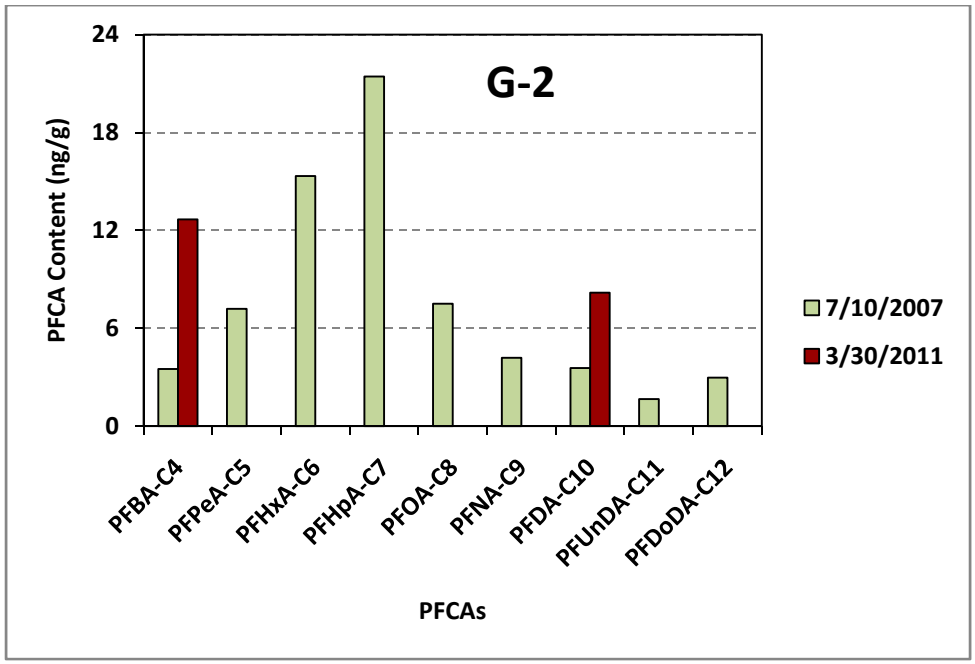

Figure 5-24. Trends of PFCAs detected in household floor wax 2 (G-2)

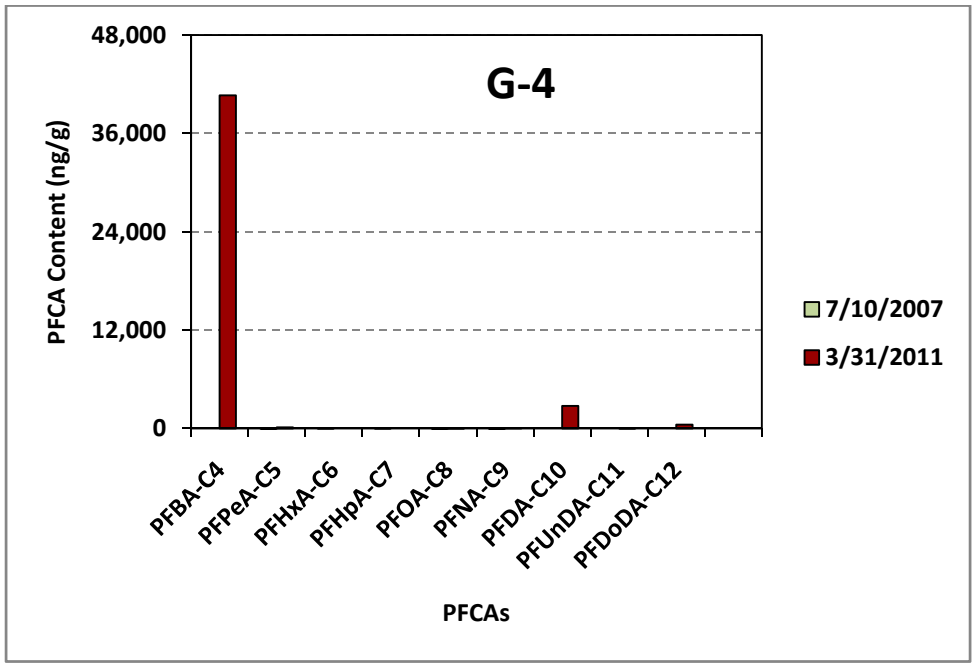

Figure 5-25. Trends of PFCAs detected in commercial floor wax 1 (G-4)

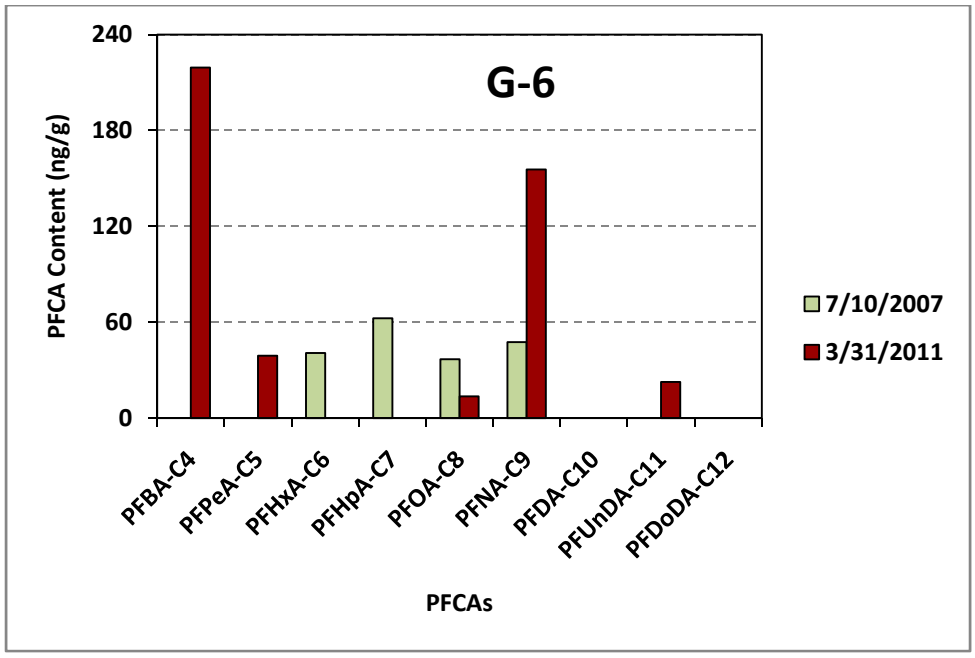

Figure 5-26. Trends of PFCAs detected in commercial floor wax 2 (G-6)

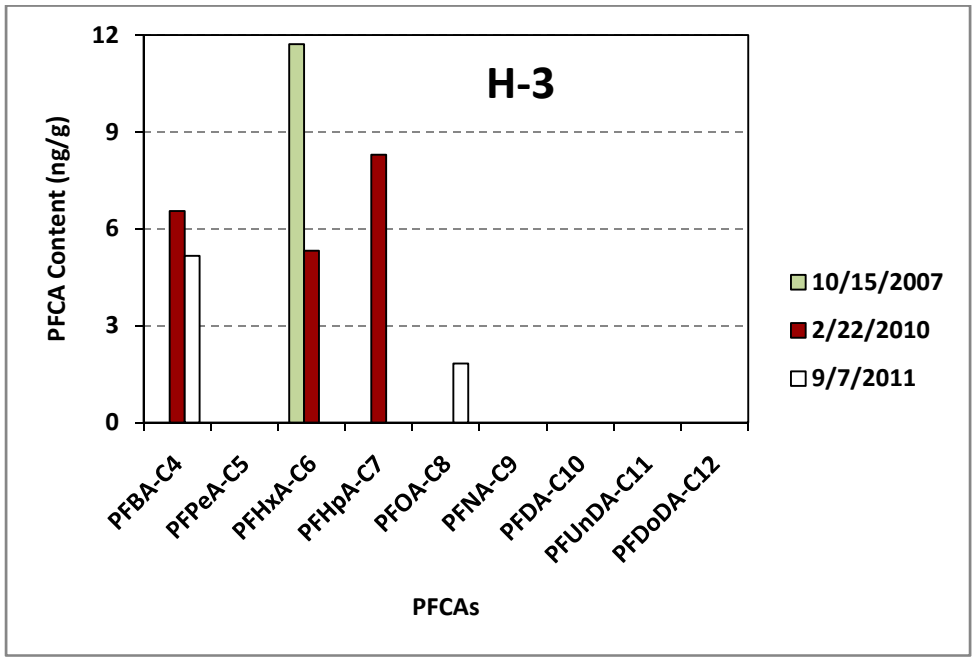

Figure 5-27. Trends of PFCAs detected in food paper 1 (H-3)

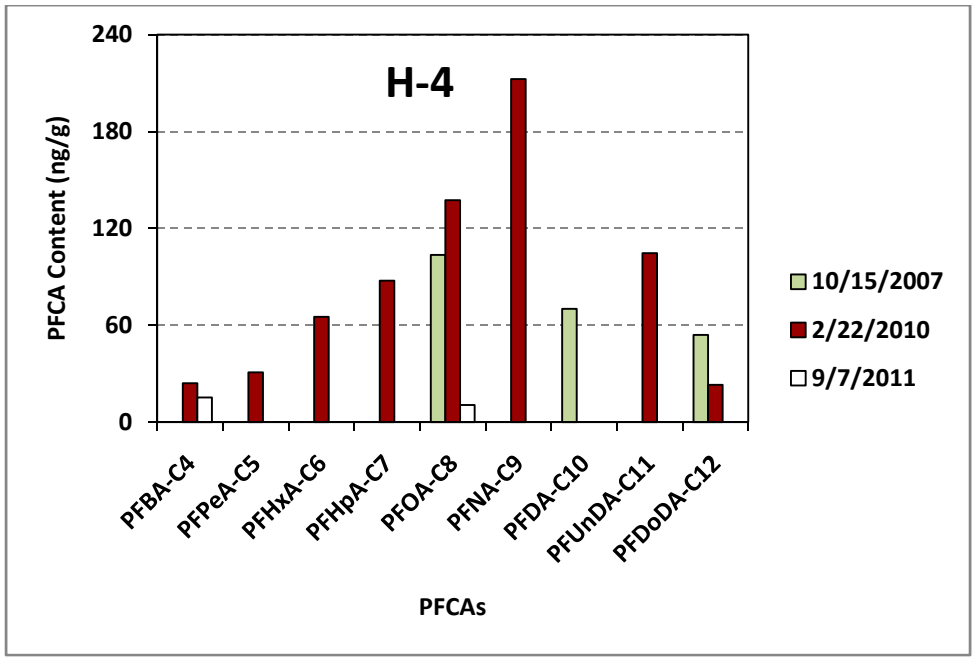

Figure 5-28. Trends of PFCAs detected in food paper 2 (H-4)

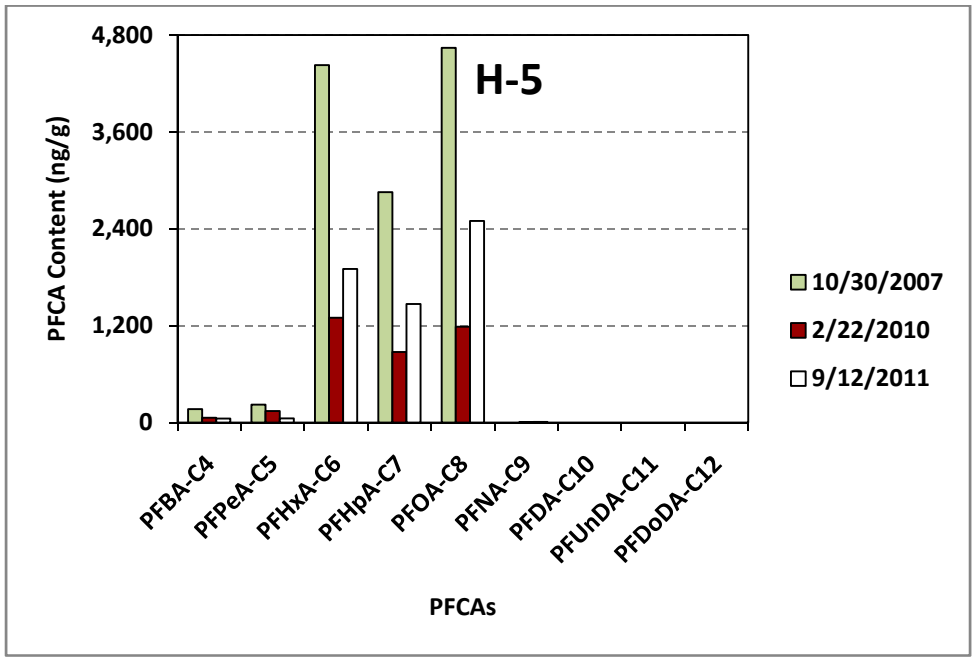

Figure 5-29. Trends of PFCAs detected in food paper 3 (H-5)

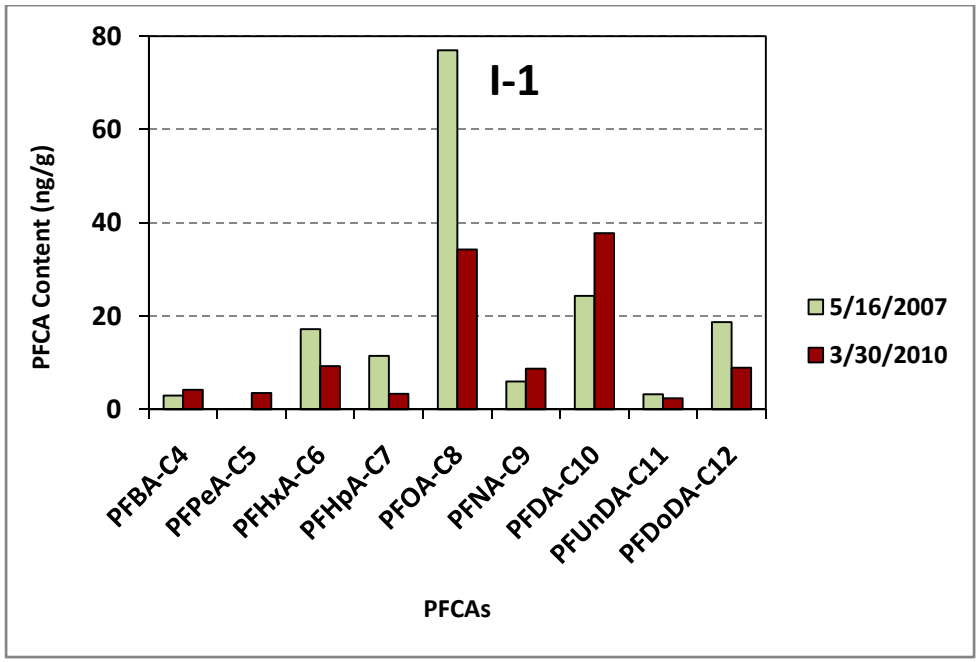

Figure 5-30. Trends of PFCAs detected in membrane 1 (I-1)

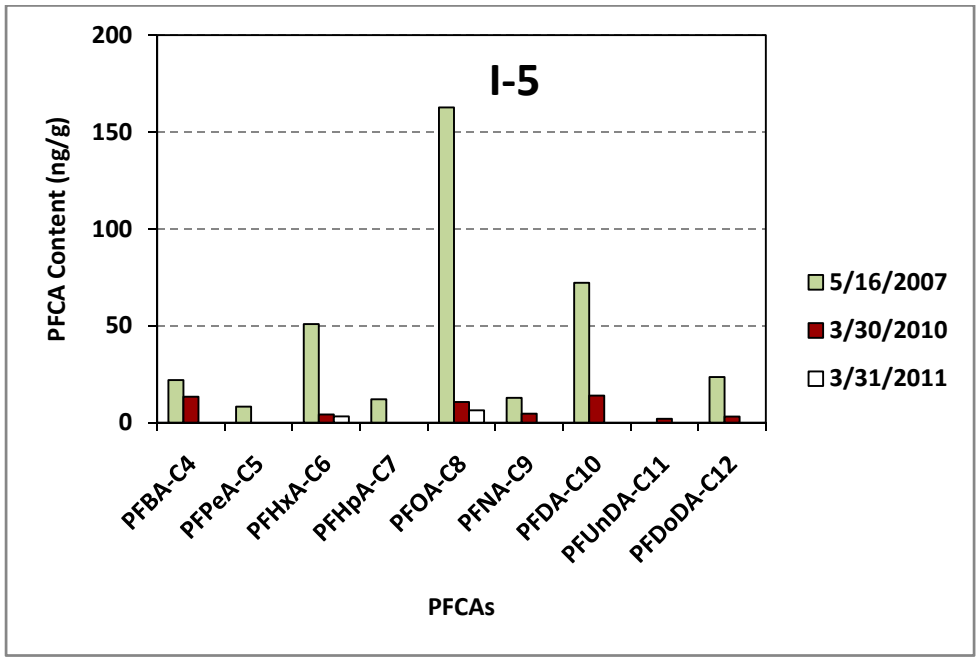

Figure 5-31. Trends of PFCAs detected in membrane 2 (I-5)

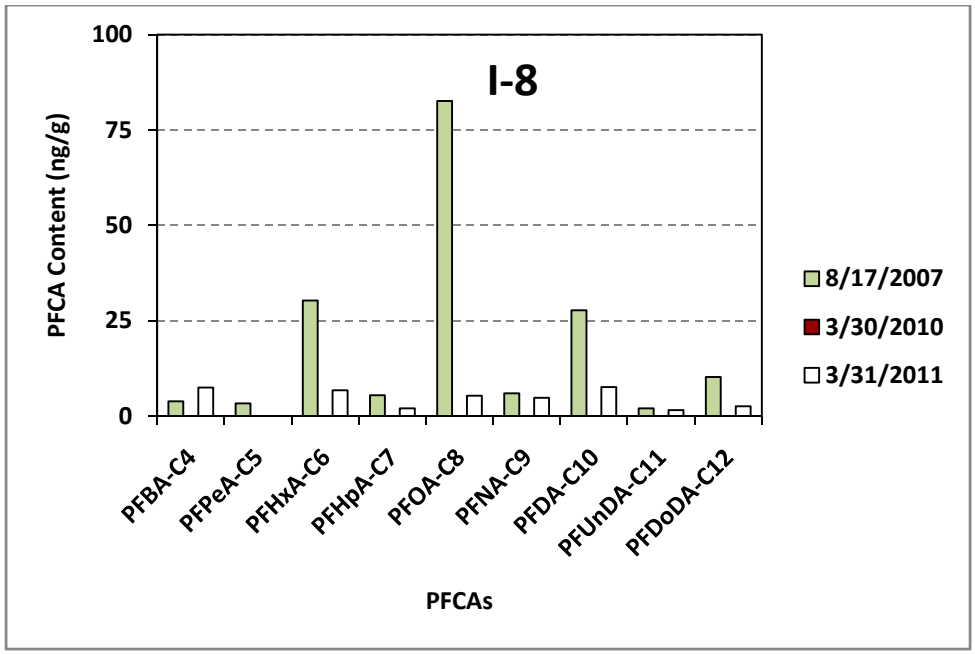

Figure 5-32. Trends of PFCAs detected in membrane 3 (I-8)

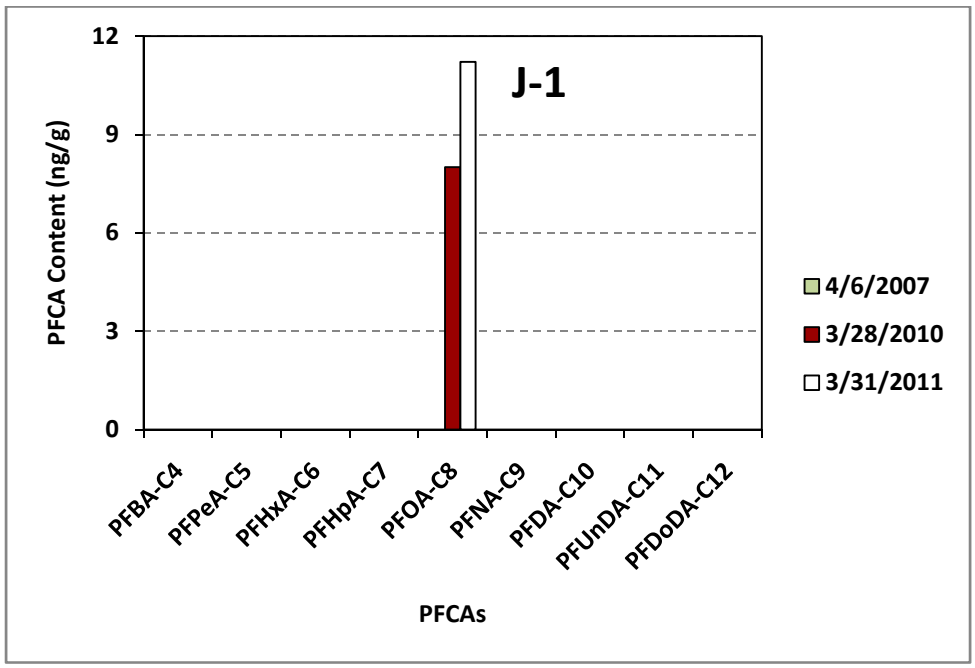

Figure 5-33. Trends of PFCAs detected in thread-sealant tape 1 (J-1)

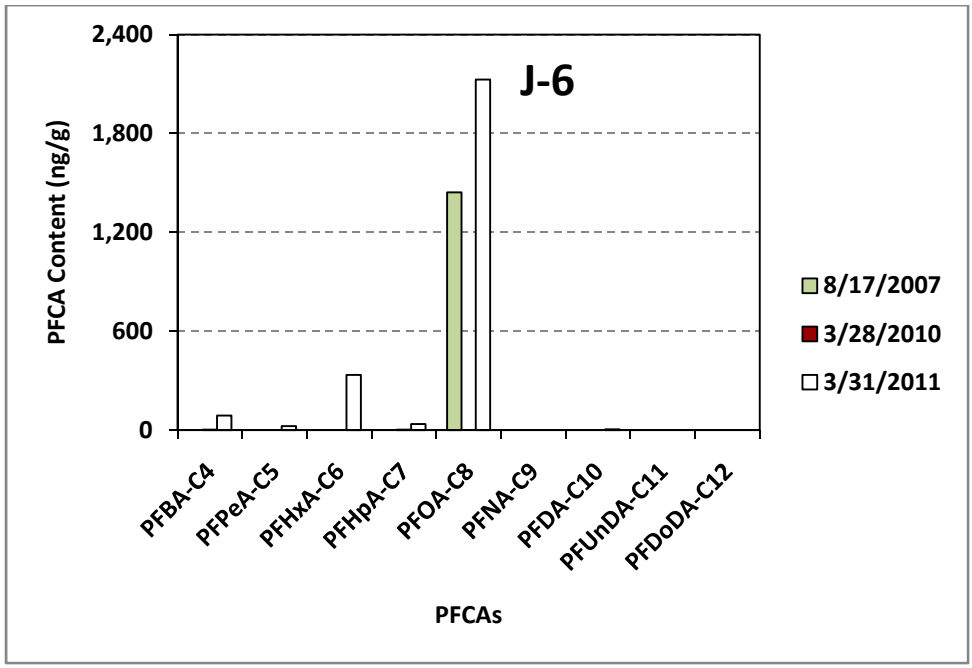

Figure 5-34. Trends of PFCAs detected in thread-sealant tape 2 (J-6)

5.3 **Total PFCAs in AOC Samples**

The total perfluorocarboxylic acids (TPFCA) is defined as the sum of the concentrations of C4 to C12, C6 to C12 or C8 to C12 PFCAs. The TPFCA of C4 to C12 values are presented in Figures 5-35 to 5-44. In the figures, the sample collection dates were represented by years. All of the data are provided in Appendix A (Tables A-1 to A-10).

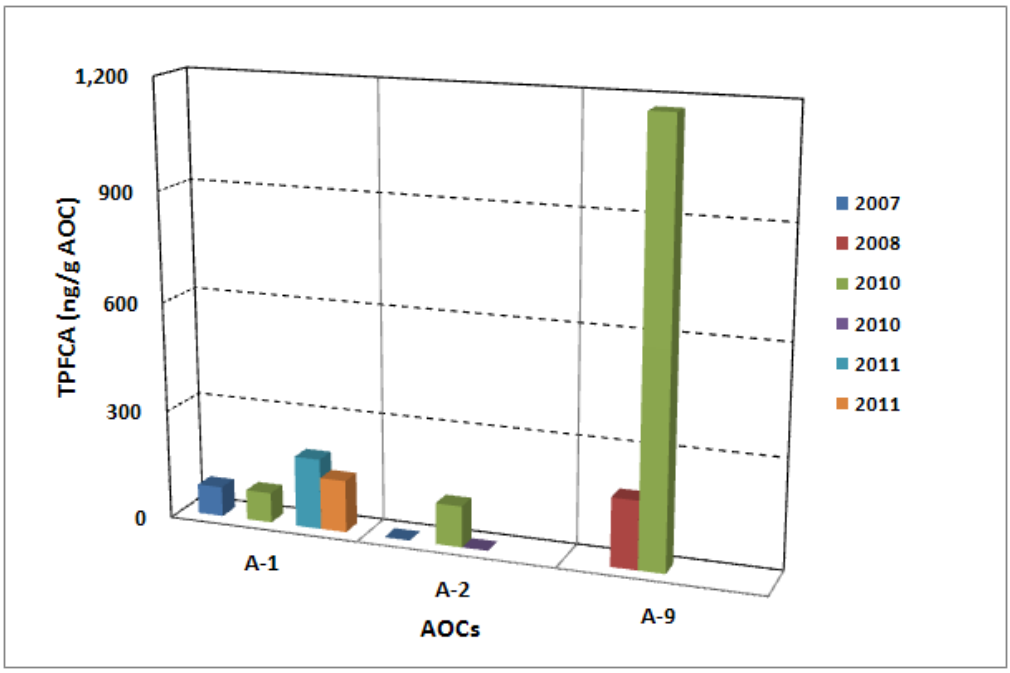

Figure 5-35. Trends of TPFCA of C4 to C12 for selected pre-treated carpeting (Two A-1 samples and two A-2 samples were collected on the same day in 2010 and 2011, respectively.)

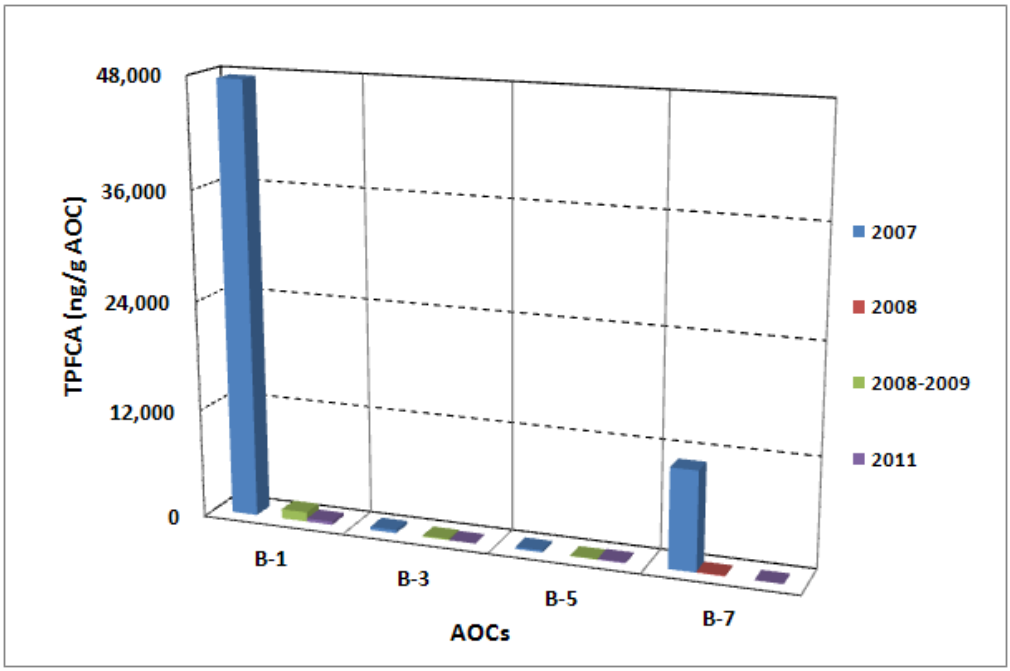

Figure 5-36. Trends of TPFCA of C4 to C12 for selected commercial carpet/fabric-care liquids (2008 is 5/2008 and 2008-2009 is 11/2008 to 5/2009)

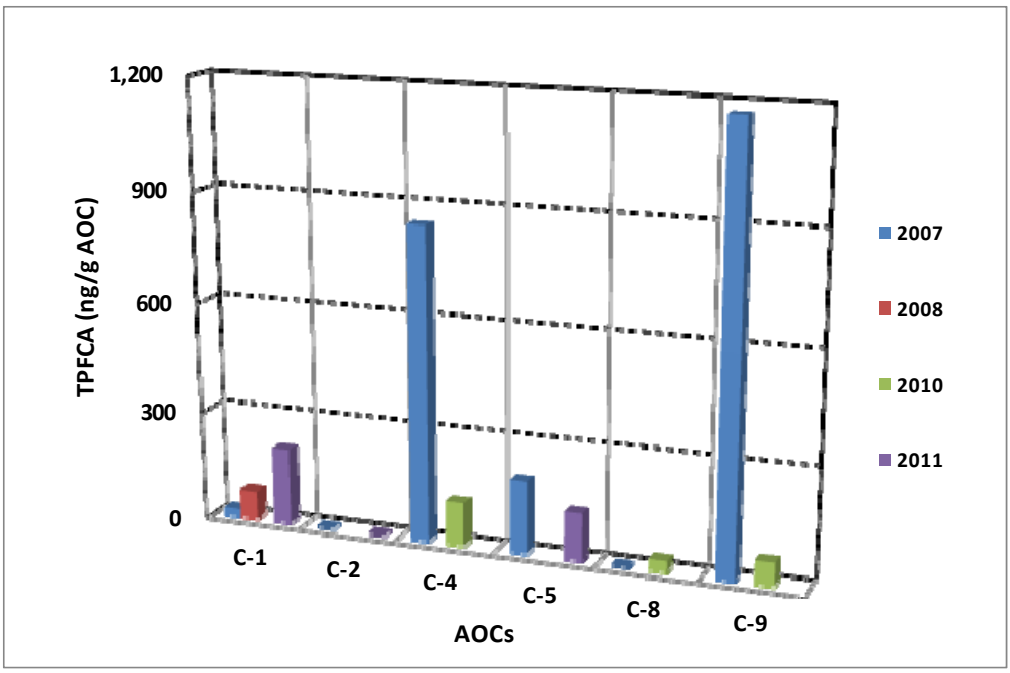

Figure 5-37. Trends of TPFCA of C4 to C12 for selected household carpet/fabric-care liquids

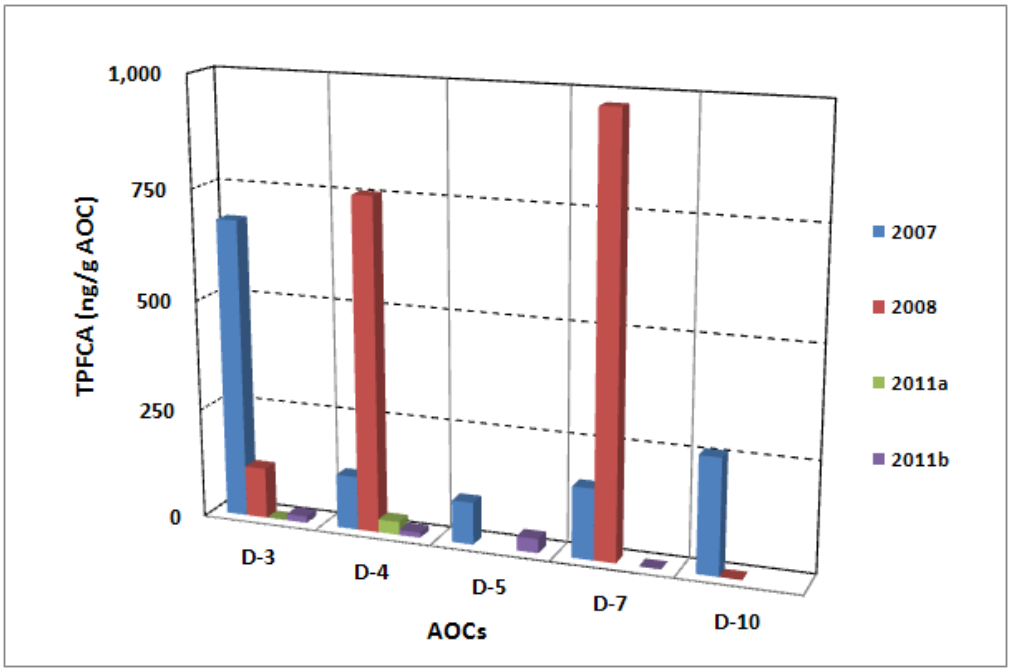

Figure 5-38. Trends of TPFCA of C4 to C12 for selected treated apparel (2011a is 2/2011 and 2011b is 8/2011)

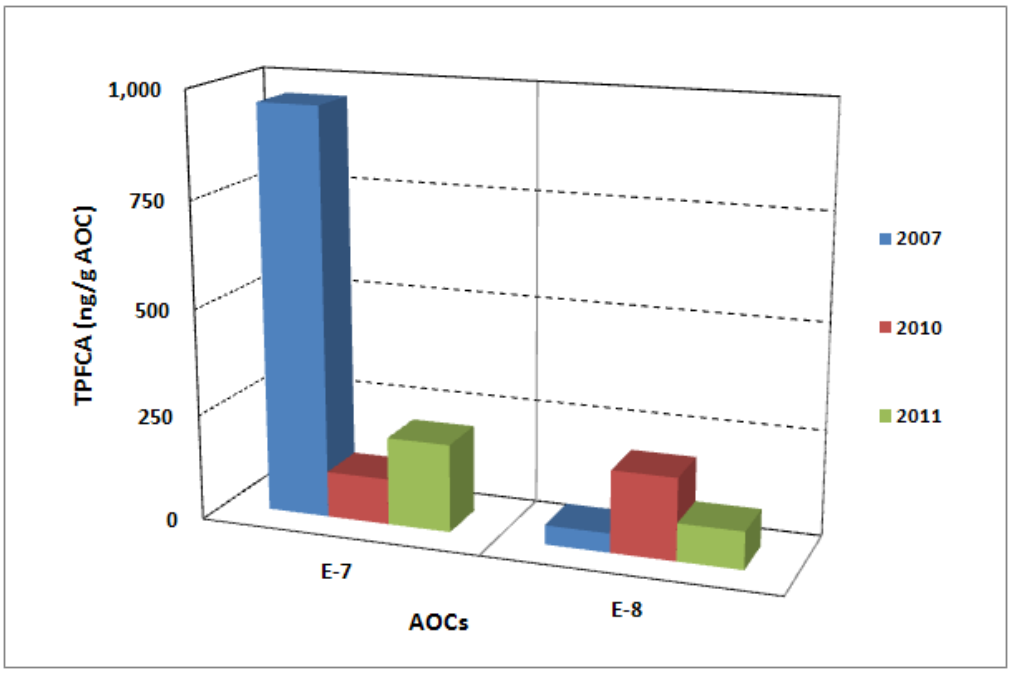

Figure 5-39. Trends of TPFCA of C4 to C12 for selected treated home textile and upholstery products

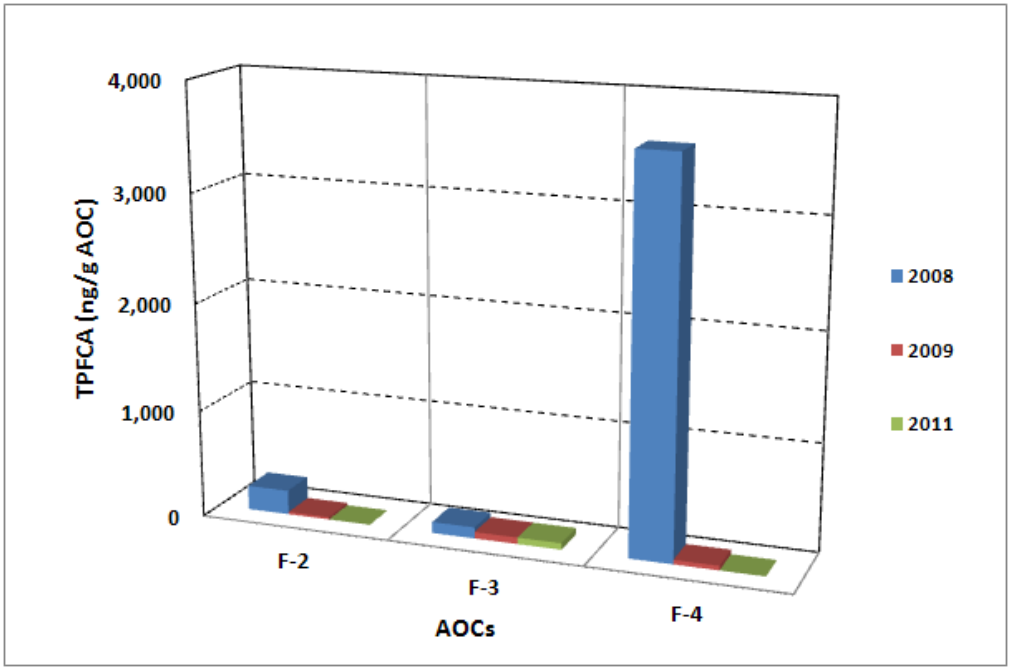

Figure 5-40. Trends of TPFCA of C4 to C12 for selected treated non-woven medical garments

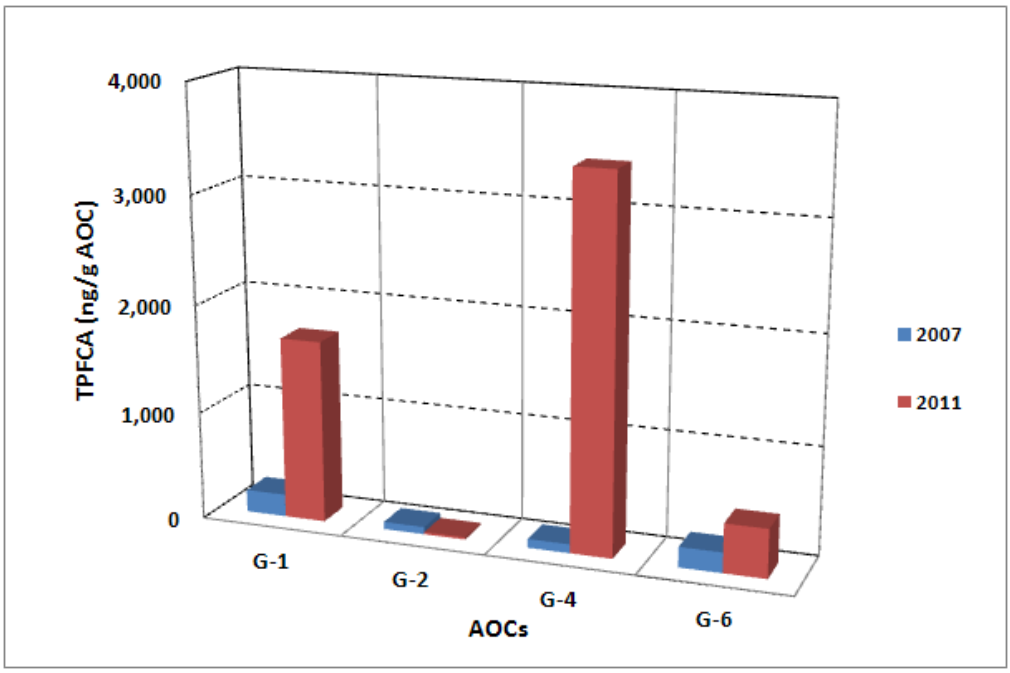

Figure 5-41. Trends of TPFCA of C4 to C12 for selected treated floor waxes and stone/wood sealants

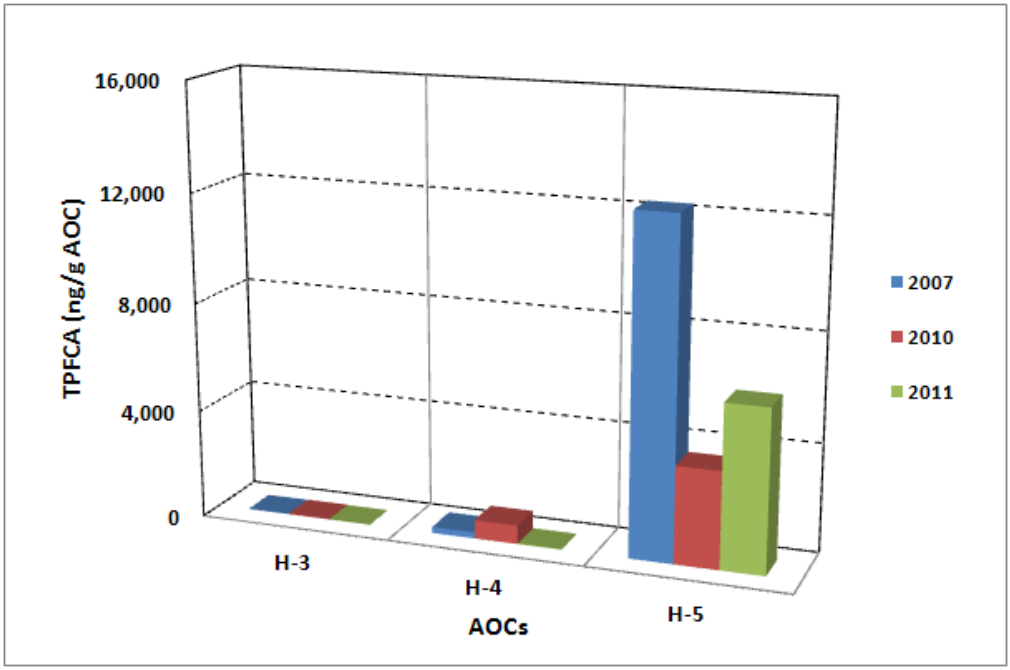

Figure 5-42. Trends of TPFCA of C4 to C12 for selected treated food contact paper

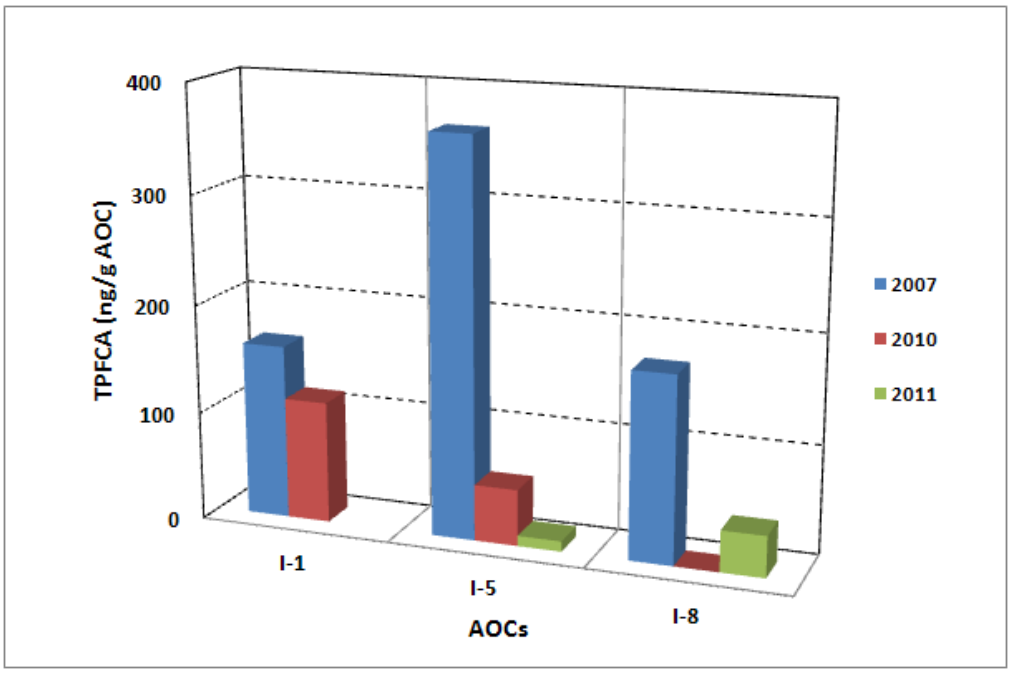

Figure 5-43. Trends of TPFCA of C4 to C12 for selected membranes for apparel

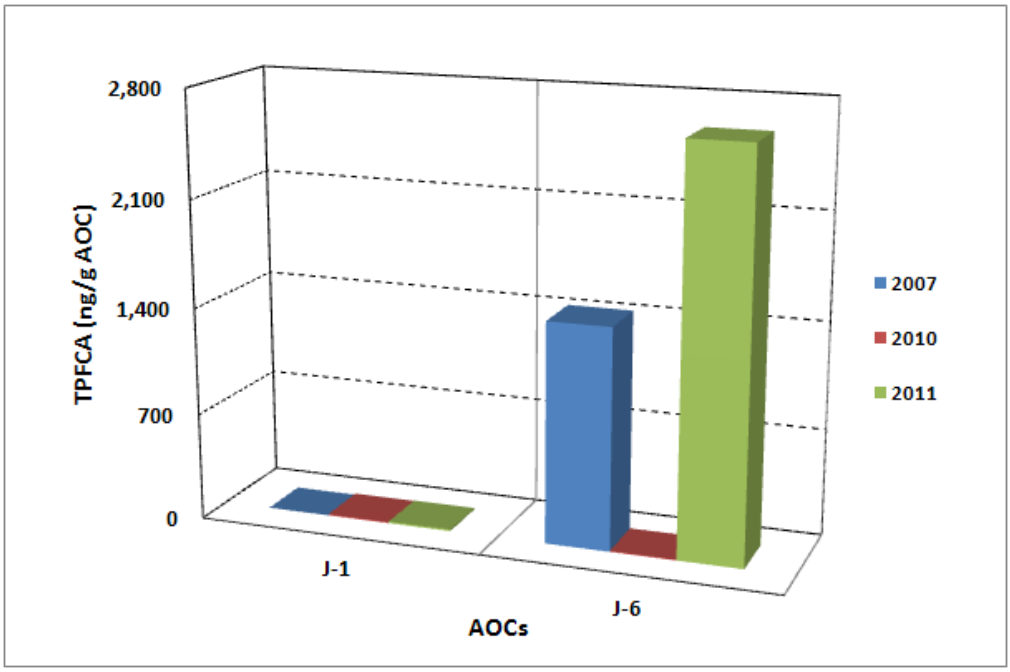

Figure 5-44. Trends of TPFCA of C4 to C12 for selected thread-sealant tapes

5.4 **Extractable PFAS Content in AOC Samples**

During the market monitoring of PFCA in AOCs, some perfluoroalkyl sulfonates were detected in the AOCs. Thus, the instrument was calibrated for PFAS, and samples that were identified as having PFAS content were reanalyzed for PFAS. The results are presented in Table 5-11. Figure 5-45 is a chromatogram that shows the detectable PFAS.

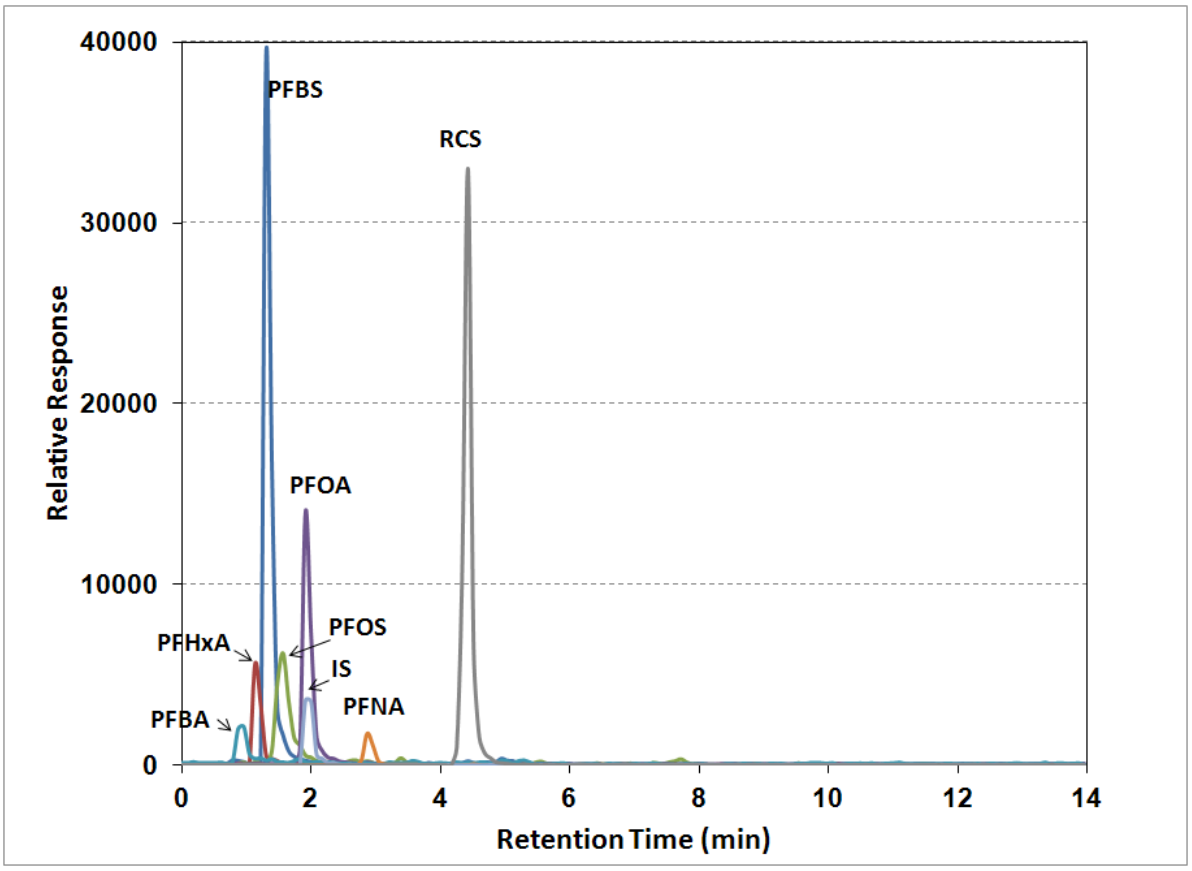

Figure 5-45. PFAS detected in one membrane for apparel (AOC I-1-1)

Table 5-11. Summary of PFAS detected in selected AOCs

AOC ID	Purchase date	PFBS-C4S	PFHxS-C6S	PFHpS-C7S	PFOS-C8S	PFDS-C10S	%RCS [a]	TPFAS [b]	N [c]
B-1-1	05/26/2009	45.8	194	190	585	19.6	80.4	1030	2
B-5-1	11/24/2008	89.6	BDL	BDL	BDL	BDL	102	89.6	2
C-1-1	11/06/2008	25.7	88.8	45.1	361	BDL	88.0	521	2
C-1-2	03/28/2011	911	155	352	257	BDL	92.2	1670	2
C-5-1	02/23/2011	161	BDL	BDL	BDL	BDL	83.3	161	2
C-9-1	03/1/2010	BDL	BDL	BDL	BDL	BDL	108	BDL	2
D-7-1	11/12/2008	2.00	BDL	BDL	BDL	BDL	109	2.00	3
D-5-2	11/12/2008	BDL	1.70	BDL	BDL	BDL	91.3	1.70	3
E-7-1	02/22/2010	BDL	BDL	BDL	BDL	BDL	119	BDL	3
E-8-1	03/30/2010	BDL	12.1	BDL	BDL	BDL	88.4	12.1	2
G-1-2	02/23/2011	143	BDL	BDL	12.0	BDL	87.0	155	2
I-1-2	03/30/2010	30.7	7.10	8.20	8.60	BDL	80.6	54.7	2
J-1-1	03/28/2010	BDL	BDL	BDL	BDL	BDL	112	112	3
J-6-1	03/28/2010	BDL	60.3	BDL	BDL	BDL	82.7	60.3	3

[a] Percent recovery of recovery check standards. [b] Total perfluoroalkyl sulfonates. [c] Number of samples analyzed for of the each AOCs.

5.5 **Data Quality**

5.5.1 LC/MS/MS Calibration

Tables 5-12 and 5-13 summarize all LC/MS/MS calibrations conducted for the project, including the lowest and the highest calibration concentrations. The linear coefficient of determination (r^2) for the calibration curve was equal to or greater than 0.991, meets the DQI goal.

The IAP results are listed in Table 5-14. The recovery percentage of IAP standards ranged from 91% to115% and %RSD of triplicate injections ranged from 0.4% to 9.5%. They all meet the criteria for IAP analysis, which were100 ± 15% recoveries and % RSD of triplicate analyses within 15%.

5.5.2 Detection Limits

The instrument detection limits were determined by three times of standard deviation of seven injections of the lowest calibration standards after each calibration. The detection limits are summarized in Table 5-15. As shown in the table, not all the instrument detection limits for LC/MS/MS met the DQI goal.

5.5.3 Quality Control Samples

The quality control samples consisted of field blanks, solvent blanks, duplicates or triplicates, daily calibration checks and recovery check standards. If the content of the analyte in the solvent blank was above the lowest calibration concentration, it was subtracted from all samples and field blanks. The data presented were not adjusted for recovery of RCS.

The RCS recoveries for individual samples are available in Appendix A and Table 5-11. More than 95 AOC samples were analyzed for the project, but only 95 samples were analyzed successfully. The data for samples that failed to meet the data quality requirements after three or more trials were discarded. The common causes of the failures were low recovery (i.e., < 80%) for the recovery check standard and poor precision of duplicate or triplicate samples (%RSD > 20%).

Table 5-16 summarizes the average recovery of DCCs for the tests. The recoveries were in a range that met the criterion for acceptable LC/MS/MS instrument performance, i.e., 85 to 115% recovery.

Table 5-12. LC/MS/MS calibration of PFCAs for the project

Date	Analyte	C4	C5	C6	C7	C8	C9	C10	C11	C12	C10-$^{13}C_2$
06/15/2011	r^2 [a]	0.9966	0.9973	0.9985	0.9995	0.9983	0.9982	0.9994	0.9995	0.9992	0.9992
	Low cal (ng/mL) [b]	0.3	0.5	0.5	0.3	0.3	0.5	0.5	0.3	0.3	0.5
	High cal (ng/mL) [c]	80	80	80	80	80	80	80	80	80	80
07/18/2011	r^2	0.9964	0.9962	0.9985	0.9988	0.999	0.9979	0.9994	0.9989	0.9987	0.9985
	Low cal (ng/mL)	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
	High cal (ng/mL)	100	100	100	100	100	100	100	100	100	100
09/13/2011	r^2	0.9929	0.9974	0.9988	0.9945	0.9972	0.9993	0.999	0.9978	0.9951	0.998
	Low cal (ng/mL)	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
	High cal (ng/mL)	100	100	100	100	100	100	100	100	100	100
10/13/2011	r^2	0.9944	0.9944	0.9971	0.9931	0.9933	0.9935	0.9923	0.9939	0.9921	0.992
	Low cal (ng/mL)	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
	High cal (ng/mL)	160	160	160	160	160	160	160	160	160	160
11/07/2011	r^2	0.9952	0.9952	0.9920	0.9931	0.9930	0.9965	0.9908	0.9937	0.9910	0.9944
	Low cal (ng/mL)	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
	High cal (ng/mL)	160	160	160	160	160	160	160	160	160	160

[a] r^2 is linear coefficient of determination. [b] Low cal is the lowest calibration concentration. [c] High cal is the highest calibration concentration.

Table 5-13. LC/MS/MS calibrations of PFAS for the project

Date	r^2 [a]		Calibration Range	
Analyte	**10/05/2011**	**11/07/2011**	**Low cal (ng/mL)[b]**	**High cal (ng/mL)[c]**
PFBS-C4	0.9977	0.9936	0.30	100
PFHxS-C6	0.9980	0.9915	0.30	100
PFHxS-C6-$^{18}O_2$	0.9997	0.9951	0.30	100
PFHpS-C7	0.9989	0.9960	0.30	100
PFOS-C8	0.9969	0.9969	0.30	100
PFDS-C10	0.9984	0.9921	0.30	100

[a] r^2 is linear coefficient of determination. [b] Low cal is the lowest calibration concentration. [c] High cal is the highest calibration concentration.

Table 5-14. IAP results for each calibration

Calibration	Analyte	IAP Concentration (ng/mL)	Avg. % Recovery	%RSD (n = 3)
06/15/2011	PFHxA-C7	11.8	113	4.5
	PFOA-C8	5.55	101	5.5
	PFDA-C10	8.09	107	6.3
07/18/2011	PFHxA-C7	11.8	97.9	0.4
	PFOA-C8	5.55	95.1	2.9
	PFDA-C10	8.09	93.3	2.7
09/13/2011	PFHxA-C7	9.87	94.2	9.5
	PFOA-C8	7.26	93.3	5.7
	PFDA-C10	9.31	105	7.9
10/05/2011	PFBS-C4	10.2	98.3	5.2
	PFHxS-C6	7.87	98.9	3.2
	PFOS-C8	9.13	115	4.6
10/13/2011	PFHxA-C7	9.87	112	6.5
	PFOA-C8	7.26	110	3.6
	PFDA-C10	9.31	110	7.5
11/07/2011	PFBS-C4	10.2	91.2	6.2
	PFHxS-C6	7.87	100	6.5
	PFOS-C8	9.13	97.7	9.4
11/07/2011	PFHxA-C7	9.87	112	3.3
	PFOA-C8	7.26	95.8	4.8
	PFDA-C10	9.31	93.1	8.6

Table 5-15. Summary of instrument detection limits (IDL, ng/mL)

Analytes	06/15/2011	07/18/2011	09/13/2011	10/13/2011	11/07/2011	Analytes	10/05/2011	11/07/2011
PFBA-C4	0.16	0.22	0.14	0.22	0.27	PFBS-C4	0.12	0.16
PFPeA-C5	0.38	0.27	0.18	0.28	0.29	PFHxS-C6	0.07	0.22
PFHxA-C6	0.35	0.18	0.25	0.29	0.28	PFHxS-C6-$^{18}O_2$	0.14	0.21
PFHpA-C7	0.23	0.29	0.17	0.28	0.28	PFHpS-C7	0.05	0 .24
PFOA-C8	0.19	0.27	0.25	0.27	0.18	PFOS-C8	0.06	0.15
PFNA-C9	0.37	0.29	0.22	0.24	0.18	PFDS-C10	0.11	0.22
PFDA-C10	0.33	0.20	0.09	0.06	0.09	--	--	--
PFUnDA-C11	0.28	0.18	0.11	0.09	0.15	--	--	--
PFDoDA-C12	0.23	0.12	0.13	0.13	0.27	--	--	--
PFDA-C10-$^{13}C_2$	0.32	0.26	0.13	0.11	0.17	--	--	--

Table 5-16. Average recoveries of DCCs for the project

Analytes	Avg. % Recovery	Std. Dev[a]	%RSD[b]	N [c]
PFBA-C4	92.7	0.08	8.4	40
PFPeA-C5	98.7	0.08	8.3	40
PFHxA-C6	96.4	0.09	9.0	40
PFHpA-C7	94.4	0.07	7.7	40
PFOA-C8	98.4	0.05	4.9	40
PFNA-C9	102	0.07	6.7	40
PFDA-C10	104	0.07	6.4	40
PFUnDA-C11	104	0.08	7.4	40
PFDoDA-C12	98.7	0.08	7.6	40
PFDA-C10-$^{13}C_2$	101	0.08	7.8	40
PFBS-C4	103	0.02	2.1	6
PFHxS-C6	112	0.03	2.7	6
PFHxS-C6-$^{18}O_2$	107	0.06	5.7	6
PFHpS-C7	111	0.02	2.1	6
PFOS-C8	101	0.04	3.6	6
PFDS-C10	91.5	0.05	5.3	6

[a] Std. Dev. is standard deviation.
[b] %RSD is percentage relative standard deviation.
[c] N is the number of DCCs analyzed.

6. Discussion

6.1 **Trends of Individual PFCAs**

Two to five AOCs from each of the ten categories were monitored for C4 to C12 PFCAs from 2007 through 2011. Depending on their market availability, products may only have been collected for two to four data points in a span of four years.

As mentioned in Section 4.1, due to the difficulty of collecting carpet samples, it was not possible to compare the PFCA concentrations between similar products. The reduction trend for PFCAs in similar products was not clear. There was still a fair amount of PFCAs in the products purchased in 2010 and 2011 relative to those purchased in 2007 to 2008.

Different temporal trends were observed for concentrations of the various PFCAs in the AOC samples. In general, the market monitoring data suggested that reduction occurred in the PFCA content in AOCs from 2007 to 2011, especially for commercial carpet/fabric-care products, treated apparel, treated non-woven medical garments, and membranes for apparel products. Significant reductions of PFOA were observed in all AOC categories except for one home textile and upholstery product and two thread-sealant tape products, in which increased PFOA concentrations were detected. It was observed that the amount of PFBA-C4 had increased in 19 of the 35 AOCs monitored, with all floor wax products showing significant increases in the amount of PFBA-C4. The results are consistent with the fact that short-chain perfluorinated compounds have become the alternatives for various uses [2, 16].

The monitoring data also showed a significant increase for most of the PFCAs in two of the treated apparel and two of the three food contact paper samples acquired in 2010 compared with those acquired in 2007 to 2008 and then followed by a decrease in samples acquired in 2011.

A non-parametric statistical method, the sign test [15], was performed using percent reduction as the null hypothesis. The sign test allocates a sign, either positive (+) or negative (-), to each observation according to whether it is greater or less than some hypothesized value [15]. The percent reduction of PFCA concentrations was calculated as:

$$Reduction\ (\%) = \left(1 - \frac{PFCA\ concentration}{Initial\ PFCA\ concentration\ measured\ in\ 2007-2008}\right) \times 100$$

The results are summarized in Table 6-1. In the table, the null hypothesis is that the percent reduction has a "+" sign when the data meet one of the following three criteria: (1) the percent reduction is greater than zero; (2) all measurements were below the detection limit; or (3) the initial concentration was not reported, but the concentrations afterwards were below the detection limit .

The "-" sign was used when the PFCA concentration was greater than its initial concentration. The table does not include pre-treated carpet products. P-values, calculated using SAS 9.2 (SAS Institute Inc., Cary, NC) were also presented in the table. A p-value < 0.05 is considered to be statistically significant, although the smaller the p-value, the stronger the evidence of a difference. The observation that there is more N+ than N- in the table and that most of the p-values are small (many less than 0.0001) provide strong evidence that the concentrations of PFCAs in AOCs have decreased. PFBA-C4 has the largest number of N- and also the largest p-value (0.14) indicating little evidence of a change in the usage of PFBA-C4, which implies the increasing relatively portion of PFBA-C4 in AOCs.

The relative abundance of each PFCA in each monitor year was calculated by:

$$Relative\ abundance\ (\%) = \left(\frac{Sum\ of\ individual\ PFCA\ in\ all\ AOCs}{Sum\ of\ All\ PFCAs\ in\ All\ AOCs}\right) \times 100$$

The relative abundances of PFCAs on the market for different years are summarized in Figure 6-1. The figure shows that PFCA C6 to C9 is predominant with PFOA (C8) being still the most abundant PFCA species on the market. It is also evident that PFBA-C4 and PFPeA-C5 had increases in the 2011 data.

Table 6-1. Sign test data and p-values for individual PFCAs detected in AOC samples

AOC ID	Purchase date	C4	C5	C6	C7	C8	C9	C10	C11	C12
B-1-1	05/26/2009	+	+	+	+	+	+	+	+	+
B-1-2	02/23/2011	+	+	+	+	+	+	+	+	NR[a]
B-3-1	11/24/2008	+	+	+	+	+	+	-	+	+
B-3-2	2/23/2011	+	+	+	+	+	+	+	+	+
B-5-1	11/24/2008	+	+	+	+	+	+	+	+	+
B-5-2	02/23/2011	+	+	-	+	+	+	+	+	+
B-7-1	05/01/2008	+	+	+	+	+	+	+	+	+
B-7-2	02/23/2011	+	+	+	+	+	+	+	+	+
C-1-1	11/06/2008	NR	+	NR	-	+	-	-	-	NR
C-1-2	03/28/2011	-	+	+	-	+	+	-	-	-
C-2-1	02/23/2011	+	+	+	+	+	+	+	+	+
C-4-1	2/22/2010	+	+	+	+	+	+	+	+	+
C-5-1	02/23/2011	NR	+	+	+	+	+	+	+	+
C-8-1	03/26/2010	NR	+	+	+	+	+	+	+	+
C-9-1	03/01/2010	-	+	+	-	+	+	+	+	+
D-3-1	11/12/2008	-	+	+	+	+	+	+	+	+
D-3-2	02/24/2011	+	+	+	+	+	+	+	+	+
D-3-3	08/16/2011	+	+	+	NR	+	+	+	+	+
D-4-1	11/24/2008	-	+	+	-	-	-	-	-	-
D-4-2	02/24/2011	-	+	+	+	+	+	+	+	+
D-4-3	08/16/2011	+	+	+	+	+	+	+	+	+
D-5-1	08/16/2011	+	+	+	NR	+	+	+	+	+
D-7-1	11/12/2008	+	+	+	-	-	-	-	-	-
D-7-2	08/16/2011	+	+	+	+	+	+	+	+	+
D-10-1	11/24/2008	+	+	+	+	+	+	+	+	+

Table 6-1. Sign test value for individual PFCAs detected in AOC samples (continued)

AOC ID	Purchase date	C4	C5	C6	C7	C8	C9	C10	C11	C12
E-7-1	02/22/2010	+	+	+	+	+	+	+	+	+
E-7-2	03/30/2011	-	+	NR	+	+	+	+	+	+
E-8-1	03/30/2010	NR	+	+	NR	-	-	-	NR	NR
E-8-2	03/30/2011	NR	+	-	NR	-	+	-	NR	NR
F-2-1	05/05/2009	+	+	+	+	+	+	+	+	+
F-2-2	03/28/2011	+	+	+	+	+	+	+	+	+
F-3-1	05/05/2009	NR	NR	NR	+	+	+	+	NR	+
F-3-2	03/28/2011	+	+	+	+	+	+	+	+	+
F-4-1	05/05/2009	NR	NR	NR	NR	+	NR	+	+	+
F-4-2	03/28/2011	+	+	+	+	+	+	+	+	+
G-1-1	02/23/2011	-	-	+	+	+	+	NR	+	+
G-2-1	03/30/2011	-	+	+	+	+	+	-	+	+
G-4-1	03/31/2011	-	+	+	+	-	-	NR	-	NR
G-6-1	03/31/2011	-	-	+	+	+	-	+	-	+
H-3-1	02/22/2010	-	+	+	-	+	+	+	+	+
H-3-2	09/07/2011	-	+	+	+	-	+	+	+	+
H-4-1	02/22/2010	-	-	NR	NR	-	-	NR	-	+
H-4-2	09/07/2011	-	+	+	+	+	+	+	+	+
H-5-1	02/22/2010	+	+	+	+	+	-	+	+	+
H-5-2	09/12/2011	+	+	+	+	+	-	+	+	+
I-1-1	03/30/2010	-	-	+	+	+	-	-	+	+
I-5-1	03/30/2010	+	+	+	+	+	+	+	NR	+
I-5-2	03/31/2011	+	+	+	+	+	+	NR	+	+
I-8-1	03/30/2010	+	+	+	+	+	+	+	+	+
I-8-2	03/31/2011	-	+	+	+	+	+	+	+	+

Table 6-1. Sign test value for individual PFCAs detected in AOC samples (continued)

AOC ID	Purchase date	C4	C5	C6	C7	C8	C9	C10	C11	C12
J-1-1	03/28/2010	+	+	+	+	-	+	+	+	+
J-1-2	03/31/2011	+	+	+	+	-	+	+	+	+
J-6-1	03/28/2010	-	+	NR	NR	NR	+	+	+	+
J-6-2	03/31/2011	-	-	NR	NR	-	+	-	+	+
Statistics	N+ [b]	29	47	45	40	43	43	40	43	46
	N- [c]	18	5	3	6	10	10	10	7	3
	NR	7	2	6	8	1	1	4	4	5
	P-value	0.1439	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001

[a] Not reported due to QA failure.
[b] N+ is the total number of "+" sign.
[c] N- is the total number of "-" sign.

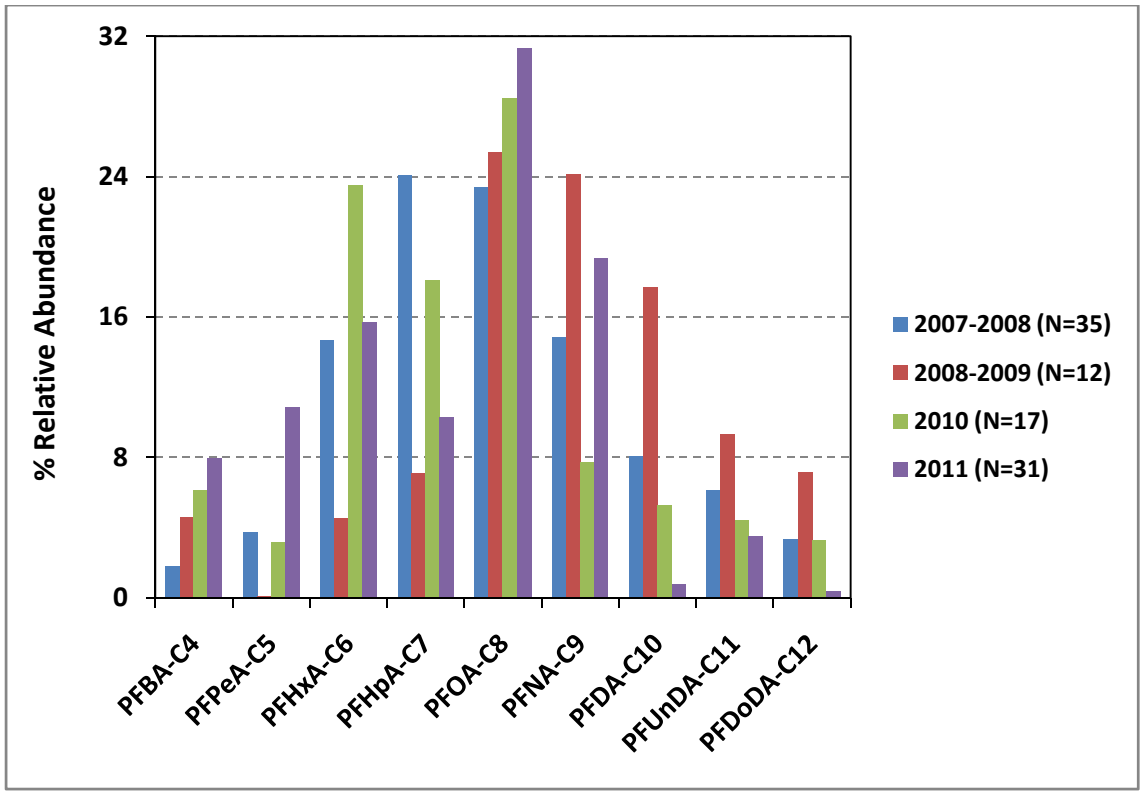

Figure 6-1. Relative abundance of PFCA in AOCs from 2007 to 2011 (N is the number of AOCs monitored)

6.2 **Trends of Total PFCAs**

The sign test results for the total PFCA (TPFCA) for C4 to C12, C6 to C12, and C8 to C12 for all monitored AOCs are presented in Table 6-2. The p-value of 0.0005 provides strong evidence that TPFCA has been reduced in a majority of the AOCs in recent years. The data presented in the tables in Appendix A also show that, among the ten categories of AOCs, commercial carpet-care liquids, treated floor waxes, treated food contact paper, and thread-sealant tapes are the most significant sources of PFCAs.

Table 6-2. Sign test results of TPFCA in AOCs

AOC Category ID	C4 to C12		C6 to C12		C8 to C12	
	N+[a]	N-[b]	N+[a]	N-[b]	N+[a]	N-[b]
B	7	1	8	0	8	0
C	4	3	6	1	6	1
D	8	2	8	2	8	2
E	2	2	2	2	2	2
F	6	0	6	0	6	0
G	1	3	1	3	1	3
H	4	2	4	2	4	2
I	5	0	5	0	5	0
J	1	3	1	3	1	3
Total	39	15	41	13	41	13

[a] Number of samples with reduced TPFCA (+). [b] Number of samples with increased TPFCA (-).

6.3 **Domestic versus Imported Articles**

It was our intention to collect domestic and imported products equally. However, since some of the imported products that we collected in Phase 1 were no longer available in the marketplace, we collected more domestic AOCs than foreign AOCs. The geographical distribution of the AOCs is presented in Figure 6-2.

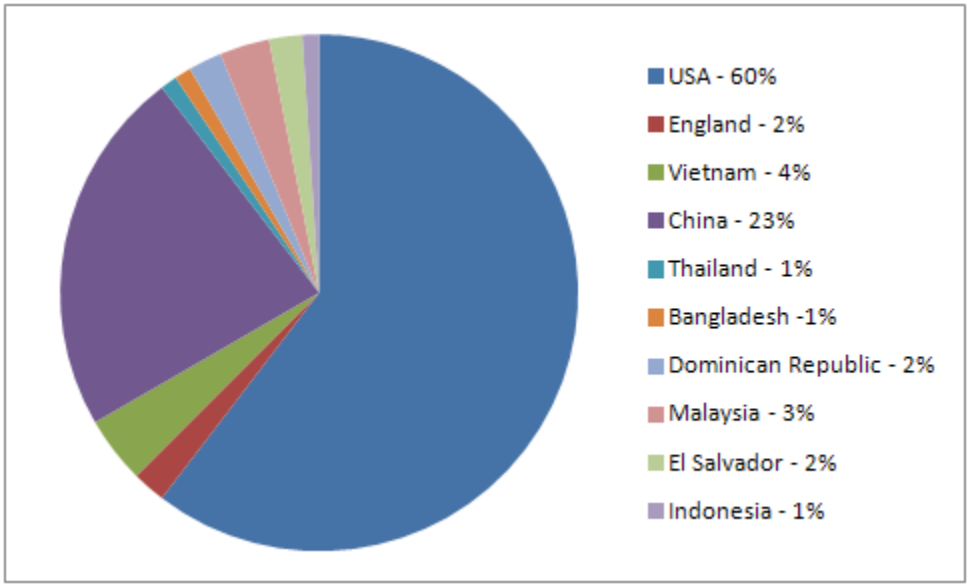

Figure 6-2. Distribution of the AOCs used for market monitoring based on country of origin

As shown in Tables 5-1 to 5-10, among the 10 article categories, all pre-treated carpet, commercial carpet-care, treated home textile and upholstery, treated floor wax, treated food contact paper, and 11 of 13 AOCs of household carpet/fabric-care products were manufactured in the United States, while treated apparel, non-woven medical garments, membranes for apparel, and thread sealant-tape products were imported. The data showed no obvious tendencies for high PFCA contents in domestic vs. imported articles.

6.4 **Short-Chain versus Long-Chain PFCAs in AOCs**

PFCA-C4 to C7 was grouped as short-chain PFCAs and PFCA-C8 to PFCA-C12 was grouped as long-chain PFCAs. The results of the sign test in Table 6-3 showed no significant difference in trends between the two groups over the monitoring period. There was not much difference between short-chain and long-chain PFCAs in AOCs in terms of N+, number of samples with reduced PFCA (+), and N-, number of samples with increased PFCA(-).

Table 6-3. Sign test results of PFCA in AOCs in short-chain group versus long-chain group

Sign Test	Short-Chain	Long-Chain
N+ [a]	161	215
N- [b]	32	40
NR [c]	23	15
P-value	<0.0001	<0.0001

[a] Number of samples with reduced PFCA (+). [b] Number of samples with increased PFCA(-).
[c] Not reported due to QA failure.

6.5 **Perfluoroalkyl Sulfonates (PFAS)**

Following the voluntary phase out of PFOS by 3M between 2000 and 2002, EPA took prompt regulatory action in 2002 to limit any future manufacture or importation of the 88 PFAS chemicals that were specifically included in the phase-out list. [13] No measurements of PFAS were conducted in Phase 1 of the project. However, we did observe PFAS during the PFCAs market monitoring study. Several AOC samples were selected for analysis to determine their PFAS C4, C6, C7, C8, and C10. Among the samples that were analyzed, carpet/upholstery protector concentrate 1, purchased in 2008, and carpet shampoo 1, purchased in 2009 and 2011, had most PFAS. The carpet shampoo product showed an increased amount of PFAS (Figure 6-3) in the products purchased in 2011 compared to the same products purchased in 2008.

It has been reported that a short-chain sulfonate (i.e., PFBS-C4), which has no bioaccumulative or toxic effects, has been developed by 3M as an alternative to PFOS. [16] The pronounced increase of PFBS-C4 in the AOC in Figure 6-3 indicates that PFBS-C4 is being used as an alternative to PFOS.

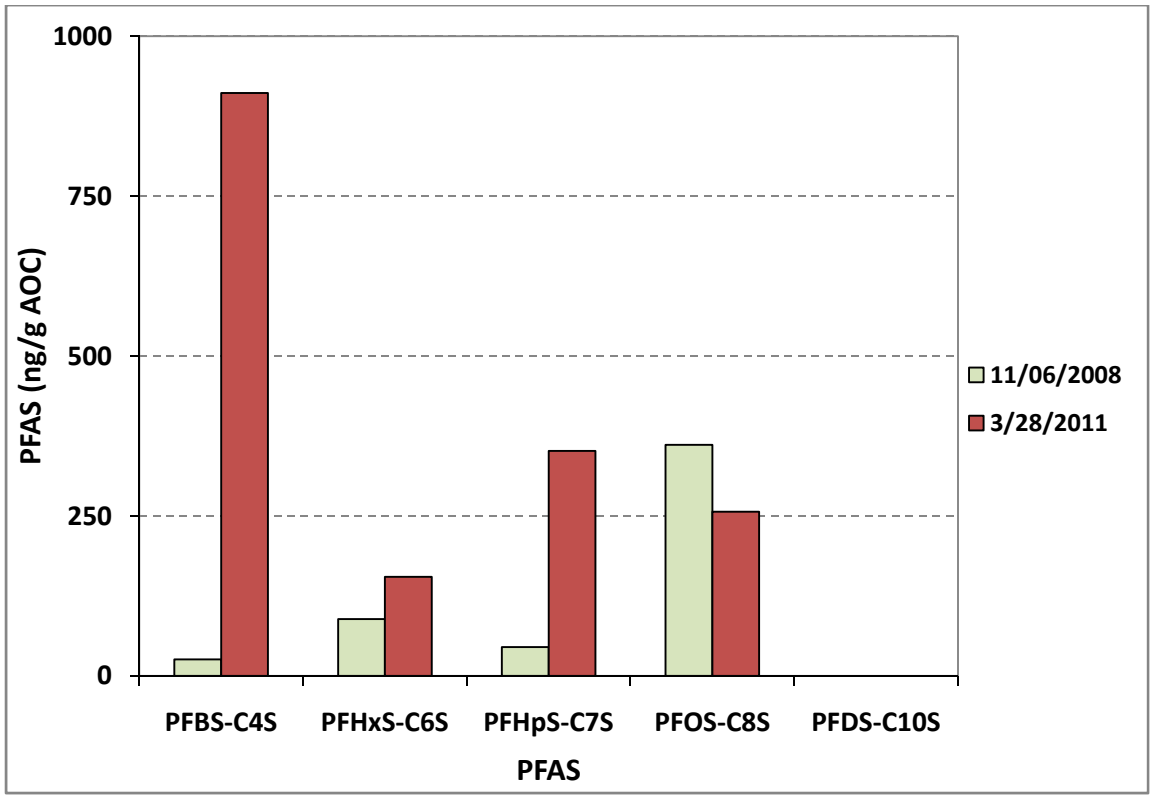

Figure 6-3. PFAS detected in a carpet shampoo in 2008 and 2011

7. References

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Appendix A. Summary of PFCAs in AOC Samples

The PFCA concentrations in AOC samples are summarized in Tables A-1 through A-11. The following abbreviations and fonts are used:

BDL = result below the detection limit;

NR = not reported (i.e., the result does not meet data quality requirements);

NA = not available, when PFBA-C4, PFPeA-C5 and perfluoroalkyl sulfonates were not calibrated in Phase 1;

RCS = recovery check standard; RCS percent recovery is the average of duplicate or triplicate samples; and

Bold Italics = result above highest calibration concentration.

TPFCA does not include data below the lowest calibration concentration or data that do not meet data quality requirements.

Table A-1. Extractable PFCAs in pre-treated carpet (ng/g AOC)

AOC ID	Purchase Date	C4	C5	C6	C7	C8	C9	C10	C11	C12	%RCS[a]	TPFCA[b]	N[c]
A-1-0	03/09/2007	4.10	NR	39.8	14.1	10.4	6.30	5.30	2.30	BDL	81.7	82.3	2
A-1-1	05/18/2010	65.1	BDL	3.70	BDL	5.50	BDL	5.20	BDL	3.40	120	82.9	3
A-1-2	09/08/2011	14.6	BDL	14.7	39.3	52.9	35.7	27.5	3.30	6.50	111	195	3
A-1-3	09/08/2011	131	BDL	8.70	BDL	3.50	BDL	BDL	BDL	BDL	107	143	3
A-2-0	03/12/2007	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	91.4	BDL	2
A-2-1	05/18/2010	107	BDL	5.80	BDL	BDL	BDL	BDL	BDL	BDL	116	113	2
A-2-2	05/18/2010	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	83.6	BDL	3
A-9-0	02/04/2008	BDL	11.5	19.2	43.0	19.9	20.7	18.4	12.3	42.0	84.7	187	2
A-9-1	05/18/2010	34.0	22.6	40.1	146	226	236	179	160	129	89.7	1172	3

[a] Percent recovery of recovery check standards. [b] Total perfluorocarboxylic acids. [c] Number of samples analyzed for each AOC.

Table A-2. Extractable PFCAs in commercial carpet-care liquids (ng/g AOC)

AOC ID	Purchase Date	C4	C5	C6	C7	C8	C9	C10	C11	C12	%RCS[a]	TPFCA[b]	N[c]
B-1-0	04/19/2007	440	1725	5197	14051	6748	8856	4376	3999	2151	101	47552	2
B-1-1	05/26/2009	67.7	BDL	61.5	118	192	311	117	109	61.8	87.5	1038	2
B-1-2	02/23/2011	186.	28.3	BDL	BDL	58.1	86.7	BDL	BDL	NR	116	359	2
B-3-0	04/19/2007	85.4	BDL	44.8	49.5	50.1	56.2	BDL	35.0	37.6	100	359	2
B-3-1	11/24/2008	BDL	BDL	BDL	8.69	38.3	44.8	12.5	7.74	BDL	85.6	112	2
B-3-2	02/23/2011	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	82.8	BDL	2
B-5-0	04/19/2007	131	BDL	30.9	19.8	19.1	BDL	BDL	BDL	19.8	99.9	220	2
B-5-1	11/24/2008	26.9	BDL	8.27	BDL	9.67	BDL	BDL	BDL	BDL	112	44.8	2
B-5-2	02/23/2011	55.7	BDL	52.9	BDL	BDL	BDL	BDL	BDL	BDL	113	109	2
B-7-0	04/19/2007	NA	363	928	2564	1838	2693	1330	844	300	101	10859	2
B-7-1	05/01/2008	BDL	BDL	22.0	BDL	25.5	17.5	21.0	16.2	BDL	101	102	2
B-7-2	02/23/2011	BDL	BDL	17.1	BDL	BDL	BDL	BDL	BDL	BDL	82.1	17.1	2

[a] Percent recovery of recovery check standards. [b] Total perfluorocarboxylic acids. [c] Number of samples analyzed for each AOC.

Table A-3. Extractable PFCAs in household carpet/fabric-care liquids (ng/g AOC)

AOC ID	Purchase Date	C4	C5	C6	C7	C8	C9	C10	C11	C12	%RCS[a]	TPFCA[b]	N[c]
C-1-0	04/19/2007	BDL	BDL	BDL	11.4	6.97	3.09	BDL	BDL	BDL	120	21.5	2
C-1-1	11/06/2008	NR	BDL	NR	13.7	BDL	37.3	9.27	16.5	NR	109	76.7	2
C-1-2	03/28/2011	94.6	BDL	BDL	19.5	BDL	BDL	15.5	48.0	28.6	120	206	2
C-2-0	05/10/2007	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	105	BDL	2
C-2-1	02/23/2011	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	84.0	BDL	2
C-4-0	05/16/2007	NA	NA	75.5	NR	666	BDL	104	BDL	BDL	98.3	846	2
C-4-1	02/22/2010	BDL	BDL	18.9	BDL	74.6	BDL	24.9	BDL	BDL	96.4	118	2
C-5-0	05/16/2007	NA	NA	195	BDL	BDL	BDL	BDL	BDL	BDL	93.4	195	2
C-5-1	02/23/2011	52.8	BDL	76.6	BDL	BDL	BDL	BDL	BDL	BDL	106	129	2
C-8-0	06/06/2007	NA	NA	BDL	BDL	BDL	BDL	BDL	BDL	BDL	81.0	BDL/NA	2
C-8-1	03/26/2010	29.8	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	81.4	29.8	2
C-9-0	09/29/2007	BDL	BDL	173	BDL	707	BDL	289	BDL	BDL	94.6	1169	2
C-9-1	03/01/2010	14.8	BDL	25.9	10.6	10.9	BDL	BDL	BDL	BDL	107	62.3	2

[a] Percent recovery of recovery check standards. [b] Total perfluorocarboxylic acids. [c] Number of samples analyzed for each AOC.

Table A-4. Extractable PFCAs in treated apparel (ng/g AOC)

AOC ID	Purchase Date	C4	C5	C6	C7	C8	C9	C10	C11	C12	%RCS[a]	TPFCA[b]	N[c]
D-3-0	05/10/2007	7.38	16.4	43.2	64.9	160.5	235	69.2	61.5	21.2	88.9	678.91	2
D-3-1	11/12/2008	36.6	BDL	12.4	12.5	15.7	22.3	8.56	4.86	2.77	119	115.59	3
D-3-2	02/24/2011	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	93.3	BDL	3
D-3-3	08/16/2011	BDL	BDL	6.20	NR	5.09	BDL	3.38	BDL	BDL	90.6	14.67	2
D-4-0	05/10/2007	6.50	NR	27.0	8.96	38.0	3.85	22.0	1.39	14.5	99.9	122.18	2
D-4-1	11/24/2008	16.8	BDL	19.1	38.8	235	164	154	47.2	79.6	95.3	755.23	2
D-4-2	02/24/2011	29.9	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	98.7	29.88	3
D-4-3	08/16/2011	BDL	BDL	8.80	BDL	2.86	BDL	BDL	BDL	BDL	99.1	11.66	2
D-5-0	05/10/2007	5.58	NR	27.2	NR	32.0	5.97	13.5	3.60	8.04	99.3	95.93	2
D-5-1	08/16/2011	BDL	BDL	4.62	2.48	4.80	2.64	8.36	3.18	6.08	119	32.16	2
D-7-0	05/10/2007	8.00	3.94	38.3	8.05	55.5	4.13	28.3	1.94	13.0	103	161.21	2
D-7-1	11/12/2008	5.18	3.51	28.2	37.7	269	199	246	98.2	87.7	120	973.75	3
D-7-2	08/16/2011	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	107	BDL	2
D-10-0	08/17/2007	5.11	NR	63.7	18.7	109	13.6	46.8	5.17	NR	96.0	261.66	2
D-10-1	11/24/2008	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	81.1	BDL	2

[a] Percent recovery of recovery check standards. [b] Total perfluorocarboxylic acids. [c] Number of samples analyzed for each AOC.

Table A-5. Extractable PFCAs in treated home textile and upholstery (ng/g AOC)

AOC ID	Purchase Date	C4	C5	C6	C7	C8	C9	C10	C11	C12	%RCS[a]	TPFCA[b]	N[c]
E-7-0	07/10/2007	17.7	21.6	68.0	96.6	330	213	125	45.7	43.0	100	961	2
E-7-1	02/22/2010	8.80	BDL	6.47	7.41	33.3	14.7	21.0	7.11	9.78	116	109	3
E-7-2	03/30/2011	116	16.44	NR	8.44	16.9	8.70	21.3	6.62	13.5	113	208	3
E-8-0	07/10/2007	NR	BDL	10.5	NR	18.8	7.15	8.99	NR	NR	106	45.5	2
E-8-1	03/30/2010	18.3	BDL	4.52	3.56	72.4	8.05	52.2	3.96	30.2	84.1	193	2
E-8-2	03/30/2011	3.48	BDL	10.6	4.97	38.1	3.80	17.8	2.73	7.24	93.6	88.8	2

[a] Percent recovery of recovery check standards. [b] Total perfluorocarboxylic acids. [c] Number of samples analyzed for each AOC.

Table A-6. Extractable PFCAs in treated non-woven medical garments (ng/g AOC)

AOC ID	Purchase Date	C4	C5	C6	C7	C8	C9	C10	C11	C12	%RCS[a]	TPFCA[b]	N[c]
F-2-0	01/30/2008	BDL	4.26	NR	18.4	47.1	82.1	20.0	24.8	8.72	86.9	205	2
F-2-1	05/05/2009	BDL	BDL	BDL	BDL	7.37	6.14	4.75	3.02	BDL	82.5	21.3	3
F-2-2	03/28/2011	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	112	BDL	2
F-3-0	01/30/2008	4.87	BDL	NR	9.03	60.7	6.33	17.4	BDL	5.30	80.4	104	2
F-3-1	05/05/2009	NR	NR	NR	8.15	37.3	4.35	9.17	NR	1.58	82.0	60.6	2
F-3-2	03/28/2011	BDL	BDL	BDL	BDL	43.5	BDL	16.6	BDL	BDL	94.0	60.1	3
F-4-0	01/30/2008	515	786	598	506	369	334	218	173	88.9	101	3588	2
F-4-1	05/05/2009	NR	NR	NR	NR	18.4	NR	9.27	7.55	6.14	94.0	41.4	2
F-4-2	03/28/2011	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	106	BDL	2

[a] Percent recovery of recovery check standards. [b] Total perfluorocarboxylic acids. [c] Number of samples analyzed for each AOC.

Table A-7. Extractable PFCAs in treated floor waxes (ng/g AOC)

AOC ID	Purchase Date	C4	C5	C6	C7	C8	C9	C10	C11	C12	%RCS[a]	TPFCA[b]	N[c]
G-1-0	07/10/2007	BDL	BDL	48.3	63.9	44.8	50.4	BDL	BDL	BDL	98.2	207	2
G-1-1	02/23/2011	45.9	1542	21.4	51.8	BDL	26.7	NR	BDL	BDL	119	1688	2
G-2-0	07/10/2007	3.50	7.19	15.3	21.4	7.50	4.19	3.56	1.65	2.97	95.4	67.3	2
G-2-1	03/30/2011	12.7	BDL	BDL	BDL	BDL	BDL	8.18	BDL	BDL	106	20.9	2
G-4-0	07/10/2007	BDL	8.62	19.1	27.3	15.6	11.8	NR	BDL	NR	116	82.3	2
G-4-1	03/31/2011	131	BDL	BDL	13.5	59.7	2737	18.4	461	BDL	98.7	3421	2
G-6-0	07/10/2007	BDL	BDL	40.7	62.5	36.9	47.6	BDL	BDL	BDL	101	187.7	2
G-6-1	03/31/2011	219	39.0	BDL	BDL	13.6	155.6	BDL	22.6	BDL	87.2	450	2

[a] Percent recovery of recovery check standards. [b] Total perfluorocarboxylic acids. [c] Number of samples analyzed for each AOC .

Table A-8. Extractable PFCAs in treated food contact paper (ng/g AOC)

AOC ID	Purchase Date	C4	C5	C6	C7	C8	C9	C10	C11	C12	%RCS[a]	TPFCA[b]	N[c]
H-3-0	10/15/2007	BDL	BDL	11.7	BDL	BDL	BDL	BDL	BDL	BDL	86.9	11.7	2
H-3-1	02/22/2010	6.55	BDL	5.32	8.29	BDL	BDL	BDL	BDL	BDL	119	20.2	3
H-3-2	09/07/2011	5.16	BDL	BDL	BDL	1.83	BDL	BDL	BDL	BDL	98.8	6.99	3
H-4-0	10/15/2007	BDL	BDL	NR	NR	104	BDL	70.2	BDL	54.0	105	228	2
H-4-1	02/22/2010	24.1	30.8	65.2	87.	137	212	NR	105	23.1	100	685	3
H-4-2	09/07/2011	15.2	BDL	BDL	BDL	10.6	BDL	BDL	BDL	BDL	92.8	25.9	3
H-5-0	10/30/2007	166	221	4427	2854	4642	BDL	BDL	BDL	BDL	103	12310	2
H-5-1	02/22/2010	59.0	142	***1298***	874	1186	5.27	BDL	BDL	BDL	93.1	3564	3
H-5-2	09/12/2011	48.7	50.9	1903	1469	2498	8.74	BDL	BDL	BDL	116	5978	3

[a] Percent recovery of recovery check standards. [b] Total perfluorocarboxylic acids. [c] Number of samples analyzed for each AOC.

Table A-9. Extractable PFCAs in membranes for apparel (ng/g AOC)

AOC ID	Purchase Date	C4	C5	C6	C7	C8	C9	C10	C11	C12	%RCS[a]	TPFCA[b]	N[c]
I-1-0	05/16/2007	2.94	BDL	17.2	11.5	77.0	5.95	24.3	3.24	18.7	109	161	2
I-1-1	03/30/2010	4.17	3.48	9.25	3.34	34.3	8.71	37.8	2.35	8.91	103	112	3
I-5-0	05/16/2007	22.0	8.23	50.9	12.1	163	12.8	72.2	NR	23.6	113	364	2
I-5-1	03/30/2010	13.4	BDL	4.22	BDL	10.6	4.65	14.0	1.98	3.11	109	51.9	2
I-5-2	03/31/2011	BDL	BDL	3.20	BDL	6.33	BDL	NR	BDL	BDL	120	9.54	2
I-8-0	08/17/2007	3.86	3.33	30.3	5.46	82.6	5.97	27.7	2.02	10.3	87.7	172	2
I-8-1	03/30/2010	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	101	BDL	3
I-8-2	03/31/2011	7.49	BDL	6.74	2.01	5.31	4.80	7.60	1.57	2.56	104	38.1	3

[a] Percent recovery of recovery check standards. [b] Total perfluorocarboxylic acids. [c] Number of samples analyzed for each AOC.

Table A-10. Extractable PFCAs in Thread sealant tapes (ng/g AOC)

AOC ID	Purchase Date	C4	C5	C6	C7	C8	C9	C10	C11	C12	%RCS[a]	TPFCA[b]	N[c]
J-1-0	04/06/2007	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	99.2	BDL	2
J-1-1	03/28/2010	BDL	BDL	BDL	BDL	8.00	BDL	BDL	BDL	BDL	81.4	BDL	2
J-1-2	03/31/2011	BDL	BDL	BDL	BDL	11.2	BDL	BDL	BDL	BDL	95.9	11.2	3
J-6-0	08/17/2007	BDL	BDL	NR	NR	1440	BDL	BDL	BDL	BDL	105	1440	2
J-6-1	03/28/2010	2.81	BDL	NR	3.35	NR	BDL	BDL	BDL	BDL	107	6.17	2
J-6-2	03/31/2011	88.6	23.8	335	37.4	2130	BDL	4.74	BDL	BDL	89.4	2620	3

[a] Percent recovery of recovery check standards. [b] Total perfluorocarboxylic acids. [c] Number of samples analyzed for each AOC.